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Effects of recycled asphalt shingle on the rheological and molecular composition properties of asphalt cement

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EFFECTS OF RECYCLED ASPHALT SHINGLE ON THE RHEOLOGICAL AND MOLECULAR COMPOSITION PROPERTIES OF ASPHALT CEMENT

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ABSTRACT

Recycling of asphalt shingles in flexible pavements has received considerable interests in recent years due to economic, environmental, and social reasons. The objective of this study is to introduce a new approach to recycle asphalt shingles in asphalt paving construction in which RAS is ground to ultra-fine particle sizes and blended with asphalt binder through a wet process. In the proposed wet process, the ground recycled material is blended with the binder at high temperature prior to mixing with the aggregates. Two unmodified binders that are classified as PG 64-22 and PG 52-28 were blended with two contrasting sources of RAS, originating from tear-off and manufacturer waste sources, at a modification content ranging from 10 to 40% by weight of the binder. The use of RAS modification through the proposed wet process was successful in the laboratory. Based on the results of the experimental program, the use of RAS modification through the proposed wet process would generally improve or not influence the high temperature grade of the binder but it may reduce the low temperature grade of the binder. An optimum shingle content may be identified that will improve the high temperature grade without influencing the low temperature grade of the binder.

Using Confocal Laser-Scanning Microscopy, wax crystals were detected. However, wax crystals were not detected in the RAS-modified binder, which may indicate that the wax molecules are absorbed by the RAS material. Results of HP-GPC showed that the proposed wet method of modification caused a slight increase of the High Molecular Weight (HMW) content in the prepared blends especially at high content of RAS modification. Use of RAS from tear-off and manufacturer waste shingles resulted in an increase in viscosity ranging from 3 to 130%. The increase in viscosity was proportional to the RAS content with greater increase at RAS content of 30% and in blends prepared with RAS from tear-off. The temperature susceptibility of the binder in the range from 95 to 135°C decreased with the use of RAS. However, the change in the binder temperature susceptibility with the use of RAS was minimal. Thixotropy and shear thinning were observed concurrently in the asphalt binder blends at 25°C. In addition, RAS-modified asphalt binders showed greater susceptibility to thixotropy than the base binder. Thixotropy increased with the increase in RAS content for both tear-off and manufacturer waste shingles. In addition, thixotropy effects were negligible at high and at low temperatures for all asphalt binder blends.

CHAPTER 1: INTRODUCTION

1.1 Background

In the United States, roads are extant in every state and region, totaling around two million miles. Remarkably, asphalt pavement represents 94 percent of these surfaces (Hansen and Newcomb 2011). In a changing world, repair and growth of this vast network must face reduction in natural sources, coupled with an increase in energy prices. These issues challenge engineers with not only a demand to save energy, but also a necessary consideration of the cost in highway construction and repair. In addition, environmental concerns must be satisfied. As a result, when the various methodologies are considered, environmental issues are tantamount, both to the users and to industry. One beneficial approach is to recycle by-product materials, which reduces consumption of virgin materials and also eliminates huge amounts of waste from being dumped into landfills. Further, the use of recycled materials should not compromise the performance of the highway. Added benefits may emerge from the recycling of by-product materials in Hot-Mix Asphalt (HMA), such as (1) reduced consumption of virgin materials; (2) reduced emissions and energy consumption during processing and manufacturing of virgin materials; (3) reduced by-product materials disposed in landfills; (4) populace, state and federal concerns over emissions; and (5) an enhanced competitiveness economically in asphalt paving construction.

One effective approach for technical, economical, and environmental aspects would be the recycling of asphalt shingles in HMA. The EPA reports that on an individual basis, the United States annually produces approximately 11 million tons of waste shingles, with the majority of it diverted to landfills. In considering this amount, ten million tons of asphalt shingles represent the results of construction and demolition (C&D) debris, while only one million tons originate from asphalt shingles manufacturers (NERC 2007). Recent studies indicate that the composition of recycled asphalt shingles includes 15 to 35% of asphalt binder. Assuming 20% asphalt binder in waste shingles, and with an outlay of 500$ for each ton of asphalt, the financial result of recycling total waste shingles may provide an annual saving of $1.1 billion, as well as strong reduction of non-renewable energy consumption in the US (Gevrenov, 2008). As an added benefit, the use of Recycled Asphalt Shingle (RAS) also allows a decrease in the amount of produced waste, as well as a positive resolution to disposal problems, especially in large cities.

Since the early 1990s, a number of research studies evaluated the use of recycled asphalt shingle (RAS) in HMA and its influence on the mix mechanical behavior. Current practices consist of dry blending RAS with the aggregates before the asphalt binder is added to the batch similar to Reclaimed Asphalt Pavement (RAP). RAS is usually ground to a uniform particle size ranging from 12.5 to 19.0 mm.

1.2 Problem Statement

Conventional practices of dry blending tear-off asphalt shingles with the aggregates before the asphalt binder is added to the batch are often criticized due to the large variability observed in the asphalt content of asphalt shingles and that the final PG grade of the binder is not known. Therefore, a new technique, known as the wet process, consisting of blending ultra-fine ground RAS with asphalt binder is proposed. In this wet process, ground recycle material is blended with original binder at high temperature prior to mixing with the aggregates, which allows for a better quality control of the chemical and physical reactions taking place in the binder blend.

1.3 Research Objectives

The objective of this study was to introduce a new approach to recycle asphalt shingles in asphalt pavement construction in which RAS is ground to ultra-fine particle sizes and blended with asphalt binder through a wet process. Laboratory tests were conducted to determine an acceptable and stable percentage of RAS that can be blended with asphalt binder. Furthermore, a combination of percentages and different RAS types were blended with virgin binders to evaluate binder performance as determined from the Superpave binder grading system. Selected combinations were also tested using advanced rheological and characterization tests.

1.4 Research Approach

The research approach adopted in this study consisted of completing the following four main tasks:

Task 1: Literature Review

A comprehensive literature review was conducted to review the following topics:

1) Types and compositions of roofing shingle;

2) Standards, suggestions and methods of using waste shingles in asphalt pavement;

3) Behavior of asphalt pavements with RAS;

4) Chemistry of asphalt binder.

Task 2: Blends Preparation

Two unmodified binders that are classified as PG 64-22 and PG 52-28 were selected (Table 1). Two contrasting sources of RAS consisting of tear off shingles from Missouri (referred to as tear-off) and manufactured shingles from Maine (referred to as manufactured) were collected from C&D processing plants. RAS materials were ground to an ultra-fine particle size distribution. Blends of asphalt binder and ground RAS were prepared at proportions of 10, 20, 30 and 40% by weight of the binder. The blends were prepared by mixing asphalt binder with RAS at a mixing temperature of 180°C using a mechanical shear mixer rotating at a speed of 1500 rpm for 30 minutes. As shown in table 1, different blends were prepared using the aforementioned test materials.

Task 3: Binder Superpave and Rheological Testing

Laboratory testing activities in this study determined the effects of RAS modification on the binder basic rheological properties, fractional compositions, and compatibility of the blends when the wet process is used. Experimental testing addressed the following important factors:

(1) Characterizing the rheological properties and molecular compositions of asphalt binders extracted from contrasting sources of RAS;

(2) Validating that the proposed process is suitable for recycling asphalt shingles in hot-mix asphalt;

(3) Determining the optimum reaction time and blending temperature for the proposed wet process; and

(4) Characterizing the rheological properties of asphalt-shingle modified asphalt binder as compared to virgin materials.

Table 1. Description of the Test Materials

Binder Abbreviation	RAS Content (%)	RAS Source	Description
Control 52	0	N/A	Conventional PG 52-28 binder with no shingle
52M10	10	Manufactured	52-28 binder with 10% RAS
52M20	20	Manufactured	52-28 binder with 20% RAS
52M30	30	Manufactured	52-28 binder with 30% RAS
52M40	40	Manufactured	52-28 binder with 40% RAS
52T10	10	Tear-off	52-28 binder with 10% RAS
52T20	20	Tear-off	52-28 binder with 20% RAS
52T30	30	Tear-off	52-28 binder with 30% RAS
52T40	40	Tear-off	52-28 binder with 40% RAS
Control 64	0	N/A	Conventional PG 64-22 binder with no shingle
64M10	10	Manufactured	64-22 binder with 10% RAS
64M20	20	Manufactured	64-22 binder with 20% RAS
64M30	30	Manufactured	64-22 binder with 30% RAS
64M40	40	Manufactured	64-22 binder with 40% RAS
64T10	10	Tear-off	64-22 binder with 10% RAS
64T20	20	Tear-off	64-22 binder with 20% RAS
64T30	30	Tear-off	64-22 binder with 30% RAS
64T40	40	Tear-off	64-22 binder with 40% RAS
SHIN	0	N/A	Conventional air-blown binder used in shingle manufacturing
EXT tear-off	0	Tear-off	Extracted binder from ground tear-off shingle
EXT manufactured	0	Manufactured	Extracted binder from ground manufactured shingle

Cigar Tube Test

The compatibility and stability of the prepared blends were evaluated using the cigar tube test (ASTM D 7173-05), which is used to determine the separation tendency of polymer-modified asphalt in the laboratory.

Superpave Binder Testing

Prepared blends were characterized using fundamental rheological tests (i.e., dynamic shear rheometry, rotational viscosity, and bending beam rheometer) and by comparing the Superpave

Performance Grade (PG) of the RAS-modified blend to the unmodified binders as per AASHTO M 320-09 (Standard Specification for Performance-Graded Asphalt Binder).

Viscosity Testing

The temperature susceptibility of the base binder and RAS-modified asphalt blends was evaluated by developing temperature-viscosity plots for the prepared samples. A Brookfield rotational viscometer was used at a test temperature ranging from 95 to 175°C according to the procedure outlined by ASTM D 4402.

Thixotropy Testing

The hysteresis technique was used to evaluate the thixotropy of the prepared blends. This method consists of subjecting the asphalt specimen to a triangular loop; i.e., a linearly increasing shear rate followed by a linearly decreasing shear rate. The area enclosed by the hysteresis loop under steady state conditions was used as a measure of thixotropy.

Dynamic Mechanical Analysis

In order to analyze the effects of RAS modification on the behavior of the binder at intermediate and high service temperatures, the dynamic mechanical functions were measured using the DSR over the entire range of temperatures and frequencies. The results were shifted in the form of master curves of complex shear moduli (G^*) and phase angles (δ).

Task 5: Molecular and Microscopic Evaluation

In order to investigate the chemical effects of RAS on asphalt binder, two different methods of testing were selected. High Pressure Gel Permeation Chromatography (HP-GPC) test was performed to measure the distribution of binder components based on molecular weight and Confocal Laser-Scanning Microscopy (CLSM) was used to determine the effects on RAS on the binder microscopic characteristics.

High Pressure Gel Permeation Chromatography (HP-GPC)

HP-GPC was conducted for a number of the prepared asphalt blends. HP-GPC presents the molecular size distribution. Fractional compositions of the binders were divided into two main groups: (1) high molecular weight (HMW), with a molecular weight of 3,000 or greater and (2) low molecular weight (LMW), with a molecular weight of 3,000 or smaller.

Confocal Laser-Scanning Microscopy (CLSM)

Microscopic analysis of the prepared asphalt blends was conducted using CLSM in a fluorescence mode. This method was selected given its ability to identify the broad fractions of asphalt binder including wax crystals. Table 2 presents a summary of number and type of tests conducted in this study.

Table 2. Details of the Experimental Program

Binder Abbreviation	**Cigar Tube**	**Superpave**	**Viscosity**	**Thixotropy**	**Dynamic Mechanical Analysis**	**GPC**
Control 52	3	2	3	3	3	2
52M10	3	2	3	3	3	2
52M20	3	2	3	3	3	_
52M30	3	_	3	_	_	_
52M40	3	2	_	_	_	2
52T10	3	2	3	3	3	2
52T20	3	2	3	3	3	_
52T30	3	_	3	_	_	_
52T40	3	2	_	_	_	2
Control 64	3	2	_	_	_	2
64M10	3	2	_	_	_	2
64M20	3	2	_	_	_	_
64M30	3	_	_	_	_	_
64M40	3	2	_	_	_	2
64T10	3	2	_	_	_	2
64T20	3	2	_	_	_	_
64T30	3	_	_	_	_	_
64T40	3	2	_	_	_	2

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

Asphalt pavement represents the most recycled and reused materials in the USA. Recycling up to 99% still fails to answer the relentless demand, as well as the cost of additives and original materials. These issues motivate highway agencies to consider applications of waste materials such as crumb rubber and asphalt shingle. Crumb rubber consists of the recycled rubber from vehicle tires. Tires are collected and then moved to a grinding device, where metals and other materials are separated from the rubber. The size of crumb rubber must be less than 1 mm (0.6 mm or passing mesh No. 40); at that point, the crumb rubber is mixed with asphalt binder through a wet process. Crumb rubber modified asphalt retains original binder properties and enhances mix resistance to rutting (Ching and Wing-gun 2006).

Shingles, on the other hand, have a structure similar to asphalt concrete. The shingles consist of aggregate and asphalt binder. Shingles are separated into two types: manufactured and tear-off shingles. Manufactured shingles are rejects that are cut off at the factory, while tear-off shingles represent waste shingles that are discarded from consumer roofs at the end of service life. Similar to crumb rubber, shingles must be free from byproducts such as wood, paper, and other unwanted materials and then ground to specific sizes. The National Asphalt Pavement Association (NAPA) reports that the use of both types of shingle waste "increased from 702,000 tons to 1.10 million tons from 2009 to 2010, a 57 percent increase." NAPA predicted that the recycling of RAS would result in saving of 234,000 tons (1.5 million barrels) of asphalt binder (Hansen and Newcomb 2011). In a comparison with crumb rubber, the use of RAS in asphalt pavement is limited to the dry method, which utilizes RAS as an aggregate, mixing it with other aggregates; this procedure comes prior to the final blending with asphalt binder. This approach causes high variability in the asphalt content of the whole mix. Further, due to the differences between shingle asphalt and the conventional asphalt used in the mix, changes in the performance grade of the mix asphalt are also observed in using RAS. These concerns may be addressed using the proposed wet process.

2.2 Shingles Production

The most common method to cover roofs is to use an individual overlapping of roof shingles. In shingle production, various materials such as wood, slate, asbestos-cement, fiber, composite or ceramics, and bitumen-soaked paper covered with aggregate (asphalt shingle), are commonly used. Two main styles of backing are used for organic and fiberglass shingle production. Organic shingle is referenced in ASTM D225-04, while fiberglass shingle is referenced in ASTM D3462-07. For each of these groups, the composition of materials is shown in table 3.

Table 3. Typical Shingle Composition (Bartlett et al. 2007)

Material	Organic Shingles	Fiberglass Shingles
Asphalt Binder	30-35%	15-20%
Aggregate	30-50%	30-50%
Fibers/Mineral Fines	15-35%	20-35%

Although different manufacturers use variable compositions, the production process remains similar. Both organic and fiberglass are impregnated with asphalt, and then coated on both sides with two different types of asphalt. One asphalt type is used as a saturte and the other is applied as a coating. Both asphalts are "air-blown" to incorporate oxygen into the asphalt for higher viscosity and stiffness. Additionally, powdered limestone (70% passing No. 200 sieve) is added as a stabilizer. The top side is surfaced with crushed rocks and granules ranging from 0.3 to 2.36 mm in order to protect against physical damage. Finally, the bottom surface is covered with fine sand (less than 0.425 mm) to prevent the individual shingles from adhering to one another during transportation (Grodinsky et al. 2002).

After production and installation, roof shingles age due to environmental factors. On a hot and sunny day, the asphalt viscosity decreases, allowing rain to gradually wash asphalt and granular aggregates away. Eventually, damaged shingles may allow water into the building. At this stage, the shingles are replaced and usually dumped as a waste material.

A major concern about waste shingle recycling is the existence of asbestos in tear-off shingles. Before 1970, asbestos was sometimes used in the manufacturing of fiberglass asphalt shingles. However, a survey of 27,000 samples tested revealed that only 1.5% of shingles contained used asbestos (Gevrenov, 2008). Another study tested 1,791 shingles for asbestos; none contained this harmful material (CMRA, 2007). The EPA disallows any materials containing greater than 1% asbestos to be used in roadway construction (Marks and Petermeier, 1997). Still, states such as Virginia require contractors to test recycled asphalt shingles for asbestos at a frequency of 1 per 100 tons before or in the middle of stockpile prior to approval (Scholz, 2010). Asbestos testing is occasionally conducted during the recycling and processing of tear-off asphalt shingles, based on the Polarized Light Microscopy (PLM) method, which detects an asbestos content of 1%. Another concern relates to the emission of polycyclic aromatic hydrocarbons (PAHs) (Gevrenov, 2008). While preliminary results show that RAS do not readily emit PAH, current research is evaluating the effect of adding discarded shingles on PAH emissions during HMA production.

2.3 Recycling Alternatives

The service life of asphalt shingles varies between 14 years in Arizona to 21 years in Pennsylvania, and is affected mainly by weather conditions. Shingles replaced after service are called tear-off shingles. Another source of waste shingles contains factory rejects and tab cut-outs. This type of shingle is called a manufactured shingle or factory scrap. The main difference between these two types is that manufactured shingles face no daily temperature fluctuations (thermal shock) or infiltration of water, while tear-off shingles contain a higher amount of asphalt, because their surface granules have been removed by the weathering process (Davis, 2009). The disposal fee for waste shingles in a landfill may reach as high as $90 to $100 per ton, depending on the location (Mallick et al., 2000).

Tear-off asphalt shingles are recycled by two methods. In the first method, tear-off shingles are separated prior to their transfer to a shingle recycling plant. In the other method, mixed roofing materials are brought to the recycling location, and then non-shingle debris is separated from the recycled material. RAS are usually processed by grinding them to a uniform particle size ranging from 12.5 to 19.0 mm. In the United States, around 10 to 11 million tons of waste asphalt shingles are produced each year (Grodinsky et al. 2002). Approximately 10 million tons of the waste shingles consist of old asphalt shingles roofing (tear-offs), while approximately

1.0 million tons of manufactured shingle asphalt are generated each year (Bartlett et al. 2007). This amount of waste attracts many markets, such as the following:

Cold patch

Filling potholes with RAS has been the practice for years in the states of New Jersey, Washington, California, and Chicago. Cold mixtures with RAS are applied in low traffic paving applications.

Dust control on rural roads

Recycled asphalt shingles were incorporated as a dust control measure on rural, granular-surfaced roadways in Iowa by applying 500 tons on 0.6 km of roadway (Marks and Petermeier, 1997). Waste asphalt shingles mixed with scrap wood were ground at a 6.6% moisture content. A magnetic roller on the discharge conveyor was used to remove most of the nails from the waste material. The ground shingles were applied on the aggregate surface, and then followed by the subsequent application of a slow-setting emulsion. The treated surface was dust-free for more than a year, displaying less vehicle noise and more service life. Based on these results, the authors concluded that the use of waste shingles as a dust control measure would save approximately $15 to $20 per ton, assuming a disposal fee of $40 per ton.

Temporary surfaces

In this application, ground RAS may be mixed with RAP and then compacted to be used as a surface for temporary roads or parking lots.

Aggregate road base and soil modification

Recent research found that adding a content of RAS ranging from 10 to 20% may reduce optimum moisture content, coupled with the strength properties of a loess soil. This process would make the blend unsuitable for soil modification with those percentages; yet a lower amount of RAS could decrease the unit weight of soil with a negligible change in strength (Rubino et al., 2005).

Production of New Shingles

A U.S. Department of Energy report determined that the addition of recycled shingles up to 20% did not affect new shingles manufacturing and also would result in energy savings. In this study, a finer ground shingle replaced the filler and some of the asphalt in new shingles (Jameson, 2008).

Fuel Source

Asphalt has an energy amount of around 20,000 BTU (British thermal unit equal to 1.055 K Joules) per pound (Mallick et al. 2000), which brings a potential to asphalt shingles for use as an energy source. This approach is mainly used in Europe, but newer studies have recently been launched in the USA (CMRA 2007). Depending on the type of shingle, organic shingles contain a BTU amount of 6 to 7.2 KBTU/lb, while fiberglass shingles have a value of 3.8 to 4.4 KBTU/lb. In a consideration of shingles as a fuel source, one main concern would be the release of asbestos in the environment at temperatures lower than 1000˚C. This concern is resolved by recent reports indicating that the percentage of asbestos in shingles is close to 0%. Another similar method concerns the application of RAS as a fuel and mineral supplement in cement kilns (Krivit, 2007). This alternative method uses organic parts of the shingle as a fuel source; any inorganic part, such as minerals and fiberglass, which do not burn, ended up as a part of the clinker. Three important benefits of this method are 1) bypassing prohibitive landfill costs for waste materials, 2) reducing the combustion energy, and 3) reducing the virgin minerals in the clinker (Krivit, 2007).

Application of RAS in pavement

Asphalt shingles are the most popular roofing materials in the US, representing approximately two-thirds of the residential roofing market (NAHB, 1998). The use of RAS in HMA is expected to provide significant benefits to the asphalt industry and highway agencies by reducing the amount of virgin asphalt binder added to the mixture. The fibrous shingle base (organic or fiberglass) also contains valuable fibers that may enhance the performance of asphalt mixtures (CMRA, 2007). Since the early 1990s, a number of research studies evaluated the use of this recycled material and its influence on the mechanical behavior of the mix. A dry blending of RAS with aggregates represents the most common use of RAS in pavement. By means of this method, RAS and aggregates are blended before asphalt binder is added to the batch, in an application matching that of RAP.

Air blown asphalt is typically used in the manufacturing of asphalt shingles; this type of asphalt binder demonstrates a greater viscosity than regular asphalt binder, used in HMA (Foo et al., 1999). Button et al. (1995) evaluated the influence of adding 5 to 10% of asphalt shingles to the mechanical properties of asphalt mixtures, compared to untreated mixes. The use of RAS resulted in a decreased tensile strength, as well as a creep stiffness of the mixture, yet it improved the resistance of the mix to moisture damage.

Gardiner et al. evaluated the influence of a manufactured and tear-off RAS content ranging from 0 to 7.5% on the mechanical properties of dense graded and stone matrix asphalt (SMA) mixtures (Gardiner et al., 1993). The use of RAS resulted in a decrease in the required virgin binder content and improved the mixture resistance to permanent deformation. Similar results were reported by other investigators (Grzybowski, 1993; Sengoz and Topal, 2004). However, mixture resistance to low temperature cracking appears to decrease when asphalt shingles are used. Ali et al. (1995) applied up to a 25% RAS in a mix and observed an increase in stiffness and resilient modulus. The mixes with RAS showed more fatigue and permanent deformation resistance, yet displayed no difference in moisture sensitivity. Foo et al. (1999) compared the properties of two HMA mixtures, prepared with conventional materials and with one source of fiberglass shingles, at a content of 5 and 10%. Results of the experimental program showed that this particular source of shingles had a high percentage of aggregates passing the 0.075 mm sieve (~35.5%). This may limit the content of asphalt shingles that can be used in the mixture performing the dry blending process. The use of asphalt shingles improved the rutting resistance of the mixture, but the mix had lower fatigue coupled with a low temperature cracking resistance. The use of RAS at a content ranging from 3 to 5% by weight of the aggregate in the preparation of Warm-Mix Asphalt (WMA) significantly improved the moisture resistance of the mixture (Xiao et al., 2011).

Maupin (2010) evaluated the use of tear-off RAS in WMA and HMA. An increase in tensile strength showed an increase in the percentage of RAS up to 5% (Maupin 2010). Zhou et al (2010) extracted shingle asphalt from RAS by means of centrifuge, and then recovered the shingle asphalt using Rotavapor Recovery. In a PG grade obtained for two manufactured and two tear-off samples, a higher temperature grade for the manufactured sample was determined to be 127˚C, and for the tear-off sample, the temperature grade proved to be above 180˚C (Zhou et al 2010).

A field evaluation of HMA, constructed of 5% manufactured shingle waste shredded to a particle size of 12.5mm, revealed an acceptable performance; the test was conducted by coring samples after 1 year and again, after 2.5 years of service (Watson et al., 1998). However, a stockpiling of RAS at the plant may cause the material to adhere in hot weather, due to its high content of asphalt binder. This concern may be addressed by using the proposed wet process. Figure 1 shows the typical steps for recycling shingles in pavement.

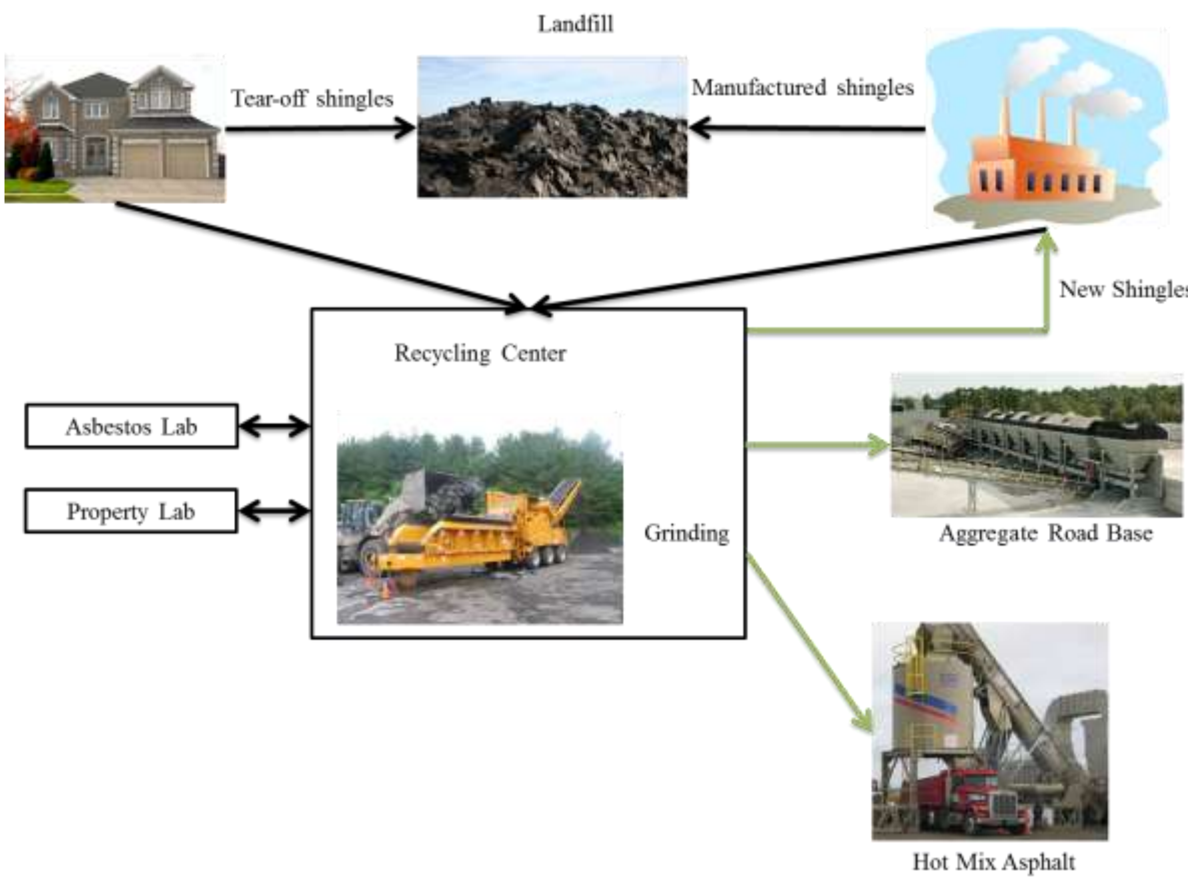


Figure 1. RAS Recycling Process and Alternatives

2.4 Standards for Use of RAS in Pavement

AASHTO MP 15-09 discusses the use of RAS in HMA as an additive, defining three types of materials: 1) manufactured shingle waste, 2) post-consumer asphalt shingle, and 3) reclaimed asphalt shingle. Manufactured shingle waste contains rejected asphalt shingles and shingle tabs from the production line. Post-costumer asphalt shingle represents the shingle taken off from a consumer's roof. Reclaimed asphalt shingle may come from both of these sources, but it must be processed to meet specifications. According to the AASHTO standard:

- All particles must be smaller than 12.5 mm (0.5 in)
- The mixture of HMA and RAS should satisfy gradation and volumetric specifications mentioned in M 323.
- If the original binder contains less than 70% of the binder in the mix (both original asphalt and shingle asphalt), then the combination of the two binders must be performance-graded to confirm the design needs.
- The RAS should be free from nails and unnecessary materials (such as metal, glass, rubber, soil, brick, tars, paper, wood, and plastic). Lightweight materials like wood, plastic and paper should be less than 1.5% by weight, with the others less than 3% retained on sieve No. 4.
- The RAS should be tested to comply with state and federal specifications for asbestos.
- The use of RAS might also alter the design criteria, as mentioned in AASHTO PP 53-09. This standard determines the amount of shingle asphalt participation in a total binder as well as considers the changes in performance grade of the total binder. A series of calculations developed in this case is as follows (AASHTO PP 53-09):

$$F_c = \frac{P_{bv} - P_{bvr}}{P_{sr}P_{br}} \qquad \Delta = P_{bv} - P_{bvr}$$

Where:

F_C = Estimated factor of shingle asphalt availability in percentage;

P_{bv} = Binder content of a mix without RAS in percentage;

P_{bvr}= Binder content of same mix with RAS in percentage;

P_{sr} = Amount of RAS used in mix in percentage;

P_{br} = Percentage of shingle asphalt in RAS;

Δ = amount of shingle asphalt working as binder in blended binder.

After two mix designs, one without RAS and the other with RAS, Δ can be calculated. Positive values of Δ show that some part of the asphalt shingle participates as the asphalt content of the mix, while the negative value shows that RAS particles are absorbing virgin asphalt and increasing the amount of virgin asphalt needed in the design. Δ depends on the quantity of shingle asphalt binder in the mix, absorption, and coating needs of RAS particles.

Furthermore, the shingle asphalt availability factor is determined as;

$$F = \frac{1+F_c}{2}$$

The amount of shingle asphalt determined to be working in the final blend is calculated below:

$$P_{brf} = \frac{F\,(P_{sr})(P_{br})}{P_{bbf}}$$

Where:

P_{brf} = percentage of shingle asphalt in total blend

P_{bbf} = amount of total binder blend in mix with RAS

At this point, performance grade of virgin binder needs to be determined so that the blend of shingle asphalt and virgin asphalt satisfy the desired performance grade. This performance grade shown below as a form of critical temperatures:

$$T_{bv} = T_{br} - \frac{T_{br} - T_{bbf}}{1 - P_{brf}}$$

Where:

T_{bv} = the critical temperature of virgin asphalt (˚C)

T_{br} = the critical temperature of shingle asphalt (˚C)

T_{bbf} = the desired critical temperature of blend (˚C)

P_{brf} = percent of shingle asphalt in total blend

In addition to the AASHTO test method, transportation agencies adopted different specifications. These regulations are summarized and are shown in table 4 for several states. Figure 2 shows the amount of RAS (tear-off or manufactured) in HMA allowed by the states.

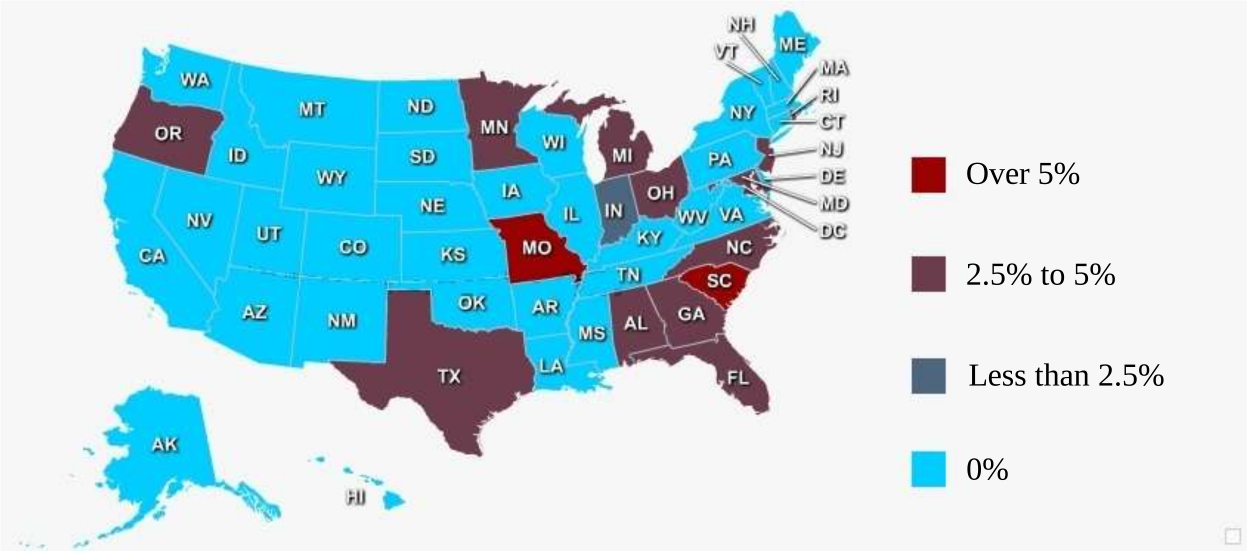


Figure 2. Allowable Percent of RAS in HMA (Scoltz, 2010 and NERC, 2012)

Table 4. Specification of Using RAS in Pavement (Scholz, 2010)

State	Standard
Alabama	100% of all dimensions must be less than 0.5 inch. • Up to 3% Tear-off RAS is allowable, by weight of aggregate. • Up to 5% Manufactured RAS is allowable, by weight of aggregate. • Up to 15% RAP and RAS for surface layers. • Up to 20% RAP and RAS for other layers. • Up to 20% RAP and RAS for plant mix bituminous base. RAS must be separated from non-shingle materials such as paper, nails, wood, and metal flashing.
Missouri	Shingle size should be less than 0.5 inch. Up to 7% Manufactured or Tear-off RAS is allowable with PG 64-22. For higher amount of RAS softer virgin binder is suggested. Up to 1.5% wood allowed in Tear-off RAS and overall 3.0% deleterious materials. Asbestos content of Stockpiles must be approved by local and national standards.

South Carolina	Shingle size should be less than 0.5 inch. 3-8% Manufactured or Tear-off RAS is allowable, by weight of aggregate. Up to 0.3% debris allowed in RAS. Must be without chemicals, oils, or any other hazardous materials (e.g., asbestos).
Texas	• 100% of all dimensions less than 0.5 inch. • Up to 5% Manufactured or Tear-off RAS allowable, by weight of mixture. • Virgin binder must contain more than 65% of the total binder for surface mixtures and 60% for other layers • Mixtures with fractionated RAP: Up to 20% RAP and RAS for surface layers. Up to 30% RAP and RAS for other layers. • Mixtures un-fractionated RAP: Up to 10% RAP and RAS for surface layers. Up to 20% RAP and RAS for other layers. Must be approved by guidelines of hazardous recyclable materials Maximum 1.5% deleterious materials allowed in stockpile

2.5 Rheological and Molecular Characterization of Asphalt Binder

2.5.1 Extraction and Asphalt Content

As mentioned earlier, the percentage of asphalt varies among RAS types. The amount and quality of asphalt used in shingle production may also vary, depending on differently producing sources. On the other hand, the longer service life of tear-off RAS might result in lower aggregate content and show higher asphalt content. Another factor is the quality of asphalt used in production, such as the air-blown asphalt in roofing shingles that are processed to gain more viscosity and stiffness. After placement of the shingles, environmental effects bring yet another aging with an oxidation process to the asphalt, which also changes asphalt characteristics.

In order to have a better understanding of shingle asphalt properties, asphalt needs to be separated from aggregates and other materials in shingle to determine first the content of asphalt and then the property of asphalt. This process is known as asphalt extraction. There are five different methods of extractions explained in ASTM D 2172-05. Quantitative analysis is the main concern in these methods. Method B, also known as Reflux Extraction, results in a separating of the asphalt with a solvent. In this method, a sample of asphalt and aggregate is placed on a net basket, while a filter paper is placed between the sample and the basket to protect materials from falling. The entire basket is placed in a jar with solvents at the bottom of the jar. A closed system is developed by using a condenser lid at the opening of the jar. Applying heat from the bottom evaporates the solvent. The solvent condenses when the lid is reached whereupon the solvent drops and washes the sample. A continuous application of this method separates the binder from the aggregates; the asphalt content may be determined by the weight difference of the basket before and after the reflux test. Furthermore, solvent may be evaporated with the application of ASTM D 5404-03. The solution is heated to a specific temperature where the solvent evaporates and then condenses in another bowl. The recovered asphalt may then be tested for its properties (Figure A1).

2.5.2 Rheological Properties

In order to understand the properties of viscoelastic materials such as asphalt binder, many experiments may be conducted. The characterization of asphalt started by chewing the asphalt, followed by tests for penetration, viscosity, and finally by tests to grade performance. The current performance grading system is based on applications of different rheological tests. New technologies can be evaluated by measuring the molecular composition of the materials and then relating those findings to the physical properties of the binder.

Viscosity

Resistance to the flow of liquids is commonly known as Viscosity. Any liquid consists of layers, which move at different velocities, viscosity appears from the accumulation of the shear stresses between the layers, resisting any applied force. Grading an asphalt binder by means of viscosity is a primary method of grading which is still regularly used. The absolute viscosity may be determined by the following equation:

$$\mu = \frac{\tau}{\gamma}$$

μ = Viscosity, τ = Shear Stress, γ = Shear Rate

The other viscosity, known as kinematic viscosity, is defined as the absolute viscosity divided by the density of the liquid at the testing temperature. Rotational viscosity is the commonly used value for grading asphalt binder. In this method, a spindle is lowered into a cylinder filled with asphalt; rotation then applies shear forces to the asphalt. Viscosity is highly dependent on the temperature of testing because normally at higher temperature there will be less resistance to shear forces. Conversely, the grading system compares viscosity measured at 135°C, due to the similarity in the manufacturing and construction temperatures. Models may be developed to show the susceptibility of materials to changes of temperature.

Superpave

Superpave performance grading (PG), a grading system for asphalt binders, relates the conditions in which a binder is used and the properties of that binder. Performance of this grading requires a consideration of environmental climate and the aging of the asphalt binder. In this regard, a series of different tests including Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) are used. The asphalt binder must pass the criteria of each test at specific temperatures and aging conditions. In this manner, an application of asphalt binder in different environments is simulated in the testing process, so the properties of these binders will show a difference. AASHTO MP 1 is used to grade asphalt binders based on the Superpave specification system.

2.5.3 Thixotropy

Thixotropy is defined as a state of change in which a thick, viscous liquid turns fluid and becomes less viscous due to shaking or agitation (Barnes 1997). This property reverses after some time in a constant condition. In another method, non-Newtonian fluids, when stirred, vibrated, or mechanically disturbed, also change to a less viscous state and return after the disturbance is stopped. Thixotropy was first identified by Peterfi in 1927 (Peterfi 1927). Pryce-Jones researched around 250 paints and noted that "It is a well-established fact that thixotropy is more pronounced in systems containing non-spherical particles" (Pryce-Jones 1936). Particles reach the best 3D structure by rotation, movement, and then change from a solid state to a thinner liquid, due to microstructural breakdown (Barnes 1997).

There are three main rheological methods to study thixotropic behavior of viscous materials: 1) the loop experiment (also known as hysteresis loop), 2) the stepwise experiment, and 3) the large amplitude oscillatory shear (LAOS) experiment. The hysteresis loop test was used in this study to characterize the thixotropic behavior of the prepared blends. In this method, the shear rate was linearly increased from zero to a constant value in a specific time period, and then was linearly decreased. In the stepwise method, stepping up or down shear rates or shear stresses are applied to the sample. The oscillatory test starts with a resting phase in which the deformation is within a linear viscoelastic area, then a high shear rotation is applied; at the last phase, amplitude and frequency returns to the resting phase. In all methods, microstructural alternation results in viscosity changes.

2.5.4 Molecular Properties

Asphalt is a viscoelastic and thermoplastic material which bonds aggregates in the asphalt pavement. The viscoelastic property of a binder changes in a response to varying compositions of asphalt binder. An evaluation of asphalt binder chemistry should be divided into two parts: 1) chemistry at the molecular level, and 2) a matrix of large molecules. The main cause of asphalt physical behavior is its composition, together with a matrix of macromolecular groups (Harrigan 1991).

At the molecular level, carbon binds with heteroatoms (sulfur, nitrogen and oxygen). The molecule becomes polar after binding, and therefore tends to react with other molecules, such as hydrogen. Different configurations of this bond result in different polar and functional groups (Jones 1992). A connection of aggregate surface and polar molecules influence strip resistance. In general, varieties of hydrocarbons contain most of the asphalt matrix. These hydrocarbons can be either aliphatic (waxy materials) or aromatic (materials seen more in air-blown asphalt), or a combination of both. Figure 3 shows the structure samples of these hydrocarbons. In figure 4, a combination of two structures is shown.

$-CH2-(CH_2)_x-CH_3$

x Typically = 15 or More Carbons

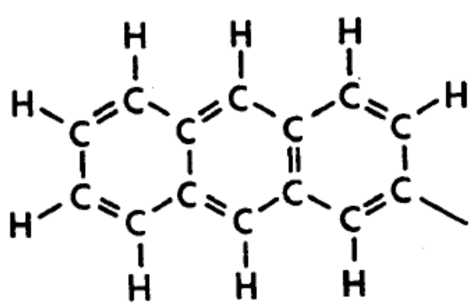


Figure 3. Aliphatic (left) and Aromatic Structure (right) (After Harrigan 1991)

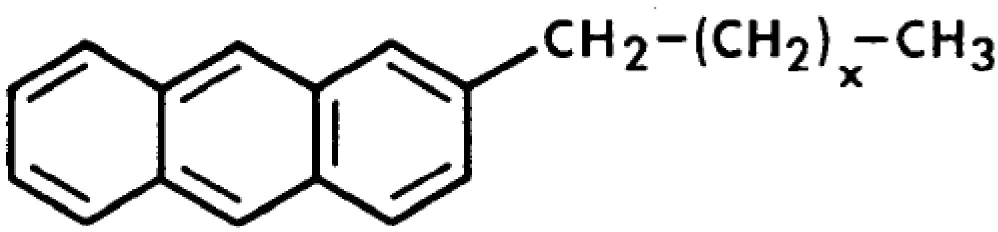


Figure 4. Mixture of Both Structures (After Harrigan 1991)

The main component of asphalt is hydrocarbon, but one or more heteroatoms may be found in most of molecules. Nitrogen sulfur, as pyridine, in the form of a benzothiophene, and oxidized carbon, similar to benzyl, are some examples of different variety of structures that can be shaped by heteroatoms. Most chemical characteristics of asphalt are described by the size of molecules. In this method, components of asphalt are categorized by solubility in different solvents. Here, asphalt can be separated into asphaltene and maltene. Maltene is soluble in n-alkane solvent, such as heptane, while asphaltene will precipitate.

Asphaltenes affect viscosity and have condensed polyaromatic hydrocarbons with large amounts of sulfur and nitrogen heteroatoms and metals (Chianelli et al. 2007). Asphaltenes have Bi- or polyfunctional molecules that contain amines, amides (source of nitrogen), as well as ketones, armides, phenols, and carboxylic acids (source of oxygen). Metals such as nickel and vanadium also are seen with nitrogen. Maltenes or petrolenes establish that part of asphalt which is soluble in pentane and heptane. These contain three main fractions, based on the Corbett method and ASTM D4124:

- Saturates are known as “materials that, on percolation in an n-heptane eluant, are not absorbed on calcined CG-20 alumina absorbent under the conditions specified” (ASTM D4214). These are saturated hydrocarbons in straight or branch chains.
- Polar aromatics or resins are lower molecular weight types of asphaltenes. Asphaltene is peptized in resins.
- Naphthene aromatics are an organic compound with $C_{10}H_8$ formula, another type of aromatic hydrocarbons with a structure similar to a pair of benzene rings.

Compositions of asphalt are shown in figure 5 in a simple way.

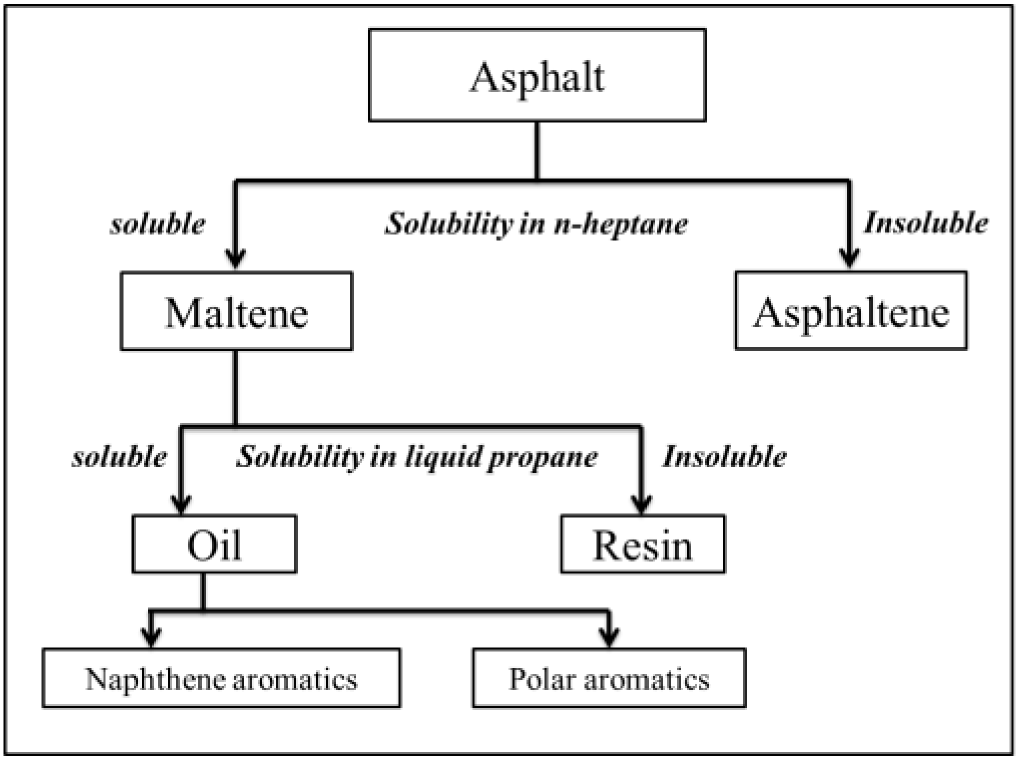


Figure 5. Compositions of Asphalt According to the Corbett method (After Harrigan 1991)

Asphaltenes are isolated and are considered to be in a dispersed phase while covered by resins, and both are surrounded in the oil phase (Jones 1992). Asphalt may be analyzed by composition by means of different techniques. Adsorption chromatography, explained in ASTM D4124, is based on solubility and adsorption, while gel permeation chromatography characterizes asphalt molecules by size. Lastly, ion-exchange chromatography separates asphalt into neutrals, and strong and weak acids, as well as strong and weak bases.

High-Pressure Gel Permeation Chromatography

High-Pressure Gel permeation chromatography (HP-GPC) analyzes asphalt molecules by size, expressed in terms of molecular weight, and volume. In this method, asphaltenes with a higher number of aromatic hydrocarbon chains are seen with higher molecular weights; on the other hand, maltenes with smaller chains have a lower molecular weight. Molecules are separated by using porous beads packed in a column. The smaller compound enters easily into the pores; thereby requiring a longer time to be released from pores. On the other hand, bigger molecules take less time on the pores and thus elute from them quickly. According to the pore openings in each column and the subsequent elution time, molecules are separated (Skoog 2006) (Figure 6).

Each column can separate a specific range of molecule size. Therefore, a proper size of pore must be determined before the test. For a high variety in size, multiple columns with different sizes are used in order to fully categorize the sample.

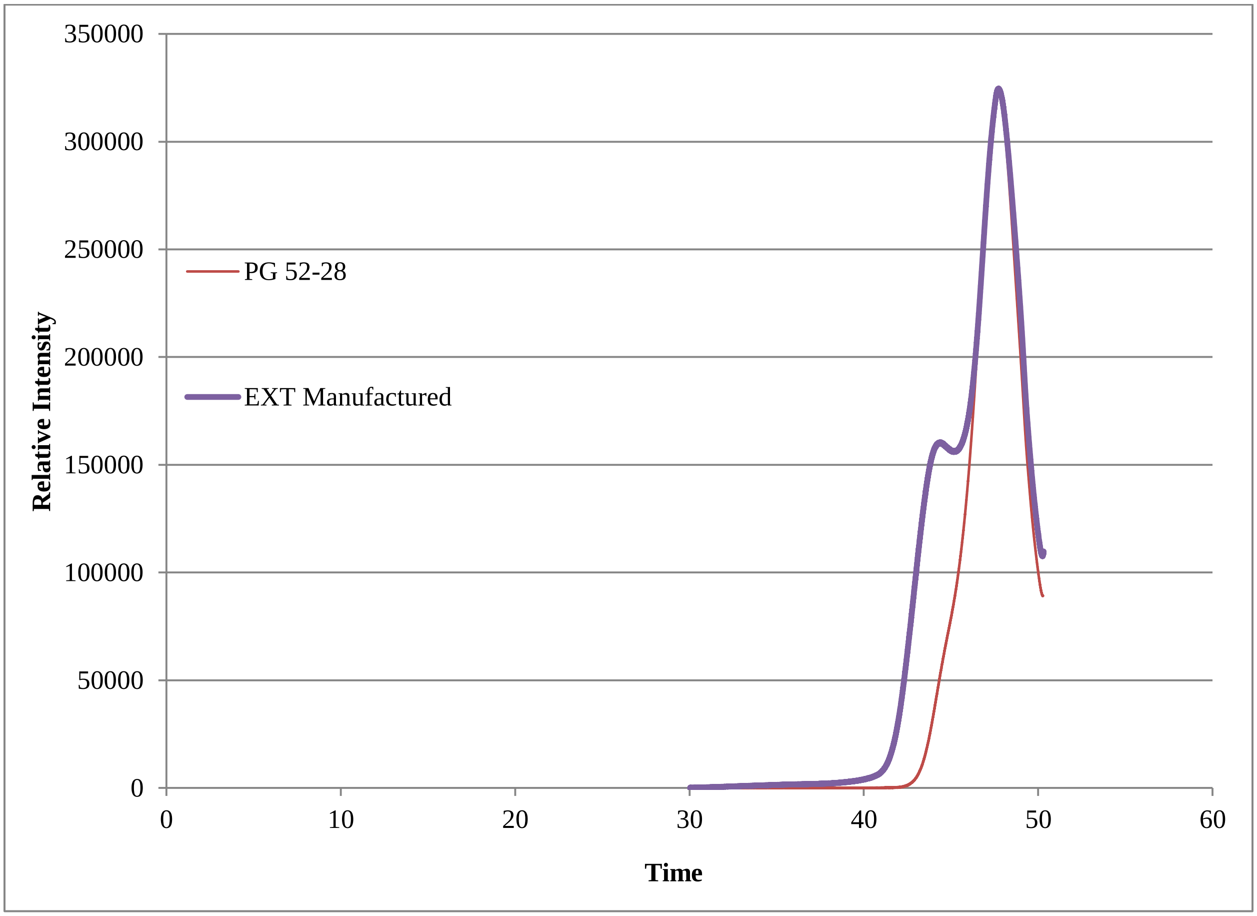


Figure 6. Sample Result of GPC

Microscopy

The microstructure of asphalt binder strongly influences its performance. Microscopic techniques may be used to observe the natural state of materials, such as asphalt binder, and thus develop a relationship between microstructure and performance in the field. Poulikakos and Partl employed environmental scanning electron microscopy (ESEM) to visualize open-graded asphalt concrete and to compare samples with the same amount of voids. Researchers detected more homogeneous air voids in polymer-modified asphalt, when comparing to control samples (Poulikakos and Partl 2010). In another study, fluorescent microscopy was used to compare the phase distribution of polymer in different polymer modified asphalt binders (Sengoz and

Isikyakar 2007). According to the images, 5 percent styrene-butadiene-styrene (SBS) was sufficient for observation of a continuous polymer phase.

Lu et al. observed waxes in an asphalt binder, with crude oils divided into waxy and naphthenic (non-waxy) oils. The study found that precipitation of wax at low temperature causes plugging in drilling, and the resulting viscosity might result in pumping problems for oil (Musser and Kilpatrick 1997). It is usually thought that crystallization of wax affects the properties of the asphalt binder. Yet, study findings detected that waxy oil exhibits a variety of micro-structures, while non-waxy oil shows no structure. The waxy oil melts at temperatures around 60˚C (Lu at al. 2005). Different structures of wax are shown in figure 7. Lu and Redelius demonstrated that although wax existence in bitumen does not affect performance at high temperature, any hardening at low temperature might increase the fracture temperature (Lu and Redelius 2007).

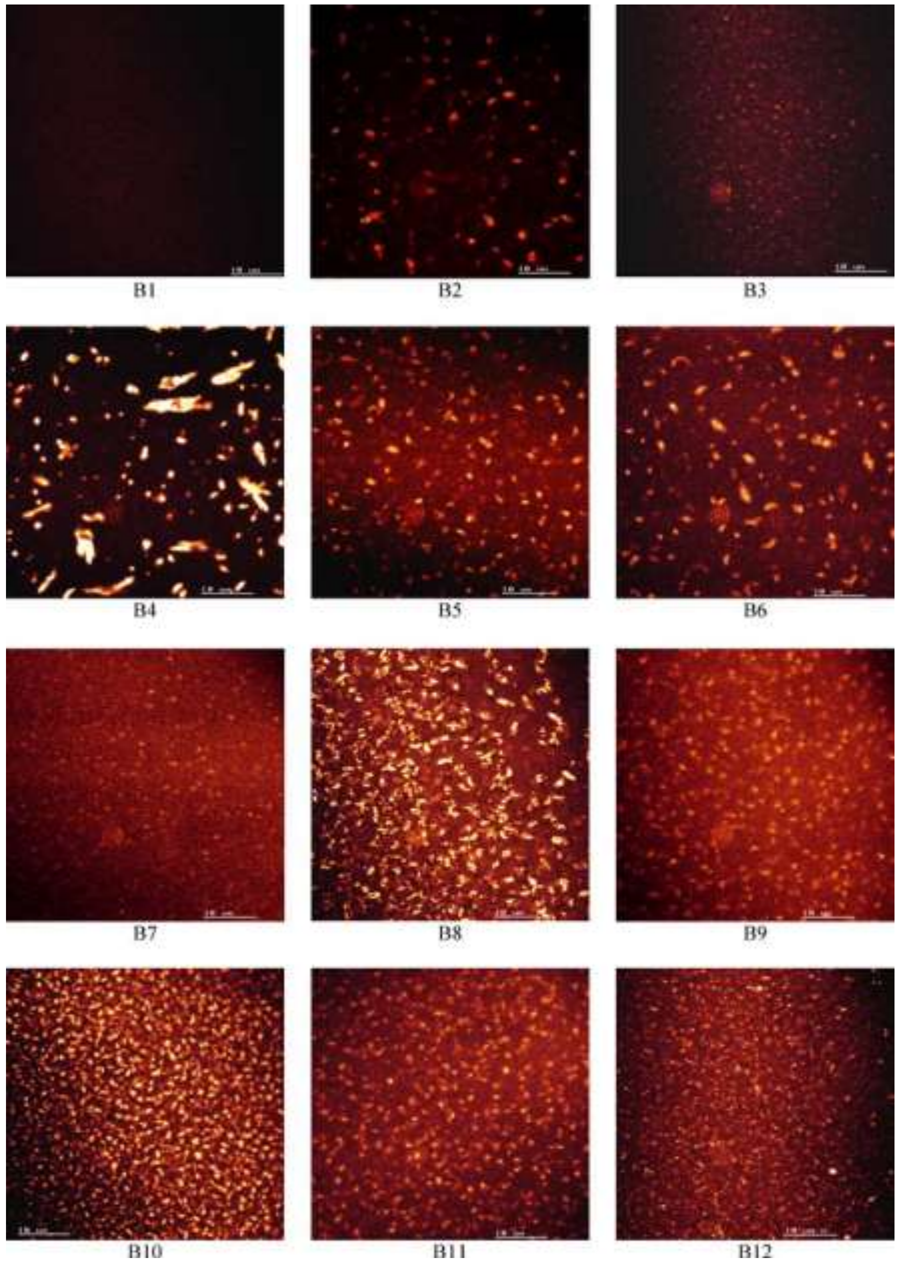


Figure 7. CLSM Micrographs of Bitumen Samples (scale: 10 μm) (after Lu et al. 2005)

CHAPTER 3: METHODOLOGY

3.1 Introduction

The experimental program included in this study was aimed at investigating the potential benefits of application of recycle asphalt shingles in asphalt paving construction. A series of laboratory tests were performed on two asphalt binders and five different content of recycled shingle. These tests designed to determine rheology and molecular characteristics of RAS added binders.

3.2 Test Materials

3.1.1 Asphalt Binders

The experimental program was designed to evaluate a wide range of asphalt blends prepared using the proposed wet process. Two unmodified binders that are classified as PG 64-22 and PG 52-28 according to the Superpave specifications were selected (Table 5). PG 64-22 is a common binder type to investigate additives in binder and PG 52-28 can indicate the effect of RAS on low temperature performance more effectively. Two contrasting sources of RAS consisting of tear off shingles from Missouri (referred to as tear-off) and manufactured shingles from Maine (referred to as manufactured) were obtained from C&D processing plants. RAS materials were ground to an ultra-fine particle size distribution at room temperature using a Pulva-Sizer® hammer mill. The utilized milling machine was equipped with a rotor assembly and hammers running at a high rotational speed of 9,600 rpm. The particle size distribution of the processed RAS was characterized using laser diffraction. The processed RAS samples were analyzed using a Beckman Coulter Particle Size Analyzer (LS13 320) operated on a wet mode. Approximately 1g of ground RAS was wetted with 26 drops of a solution of glycerol and water followed by 20 sec of bath sonication. Results of the particle size analysis using laser diffraction are presented in table 6 for the ground tear-off and manufactured RAS materials. As shown in this table, the mean particle sizes were 85.5 μm for tear-off and 201.0 μm for manufactured with a standard deviation approximately equal to the mean of the distribution indicating that the particle size distribution is heavily weighted far from the mean.

Table 5. Description of the Test Materials

Binder Abbreviation	RAS Content (%)	RAS Source	Description
Control 52	0	N/A	Conventional PG 52-28 binder with no shingle
52M10	10	Manufactured	52-28 binder with 10% RAS
52M20	20	Manufactured	52-28 binder with 20% RAS
52M30	30	Manufactured	52-28 binder with 30% RAS
52M40	40	Manufactured	52-28 binder with 40% RAS
52T10	10	Tear-off	52-28 binder with 10% RAS
52T20	20	Tear-off	52-28 binder with 20% RAS
52T30	30	Tear-off	52-28 binder with 30% RAS
52T40	40	Tear-off	52-28 binder with 40% RAS
Control 64	0	N/A	Conventional PG 64-22 binder with no shingle
64M10	10	Manufactured	64-22 binder with 10% RAS
64M20	20	Manufactured	64-22 binder with 20% RAS
64M30	30	Manufactured	64-22 binder with 30% RAS
64M40	40	Manufactured	64-22 binder with 40% RAS
64T10	10	Tear-off	64-22 binder with 10% RAS
64T20	20	Tear-off	64-22 binder with 20% RAS
64T30	30	Tear-off	64-22 binder with 30% RAS
64T40	40	Tear-off	64-22 binder with 40% RAS
SHIN	0	N/A	Conventional air-blown binder used in shingle manufacturing
EXT tear-off	0	Tear-off	Extracted binder from ground tear-off shingle
EXT manufactured	0	Manufactured	Extracted binder from ground manufactured shingle

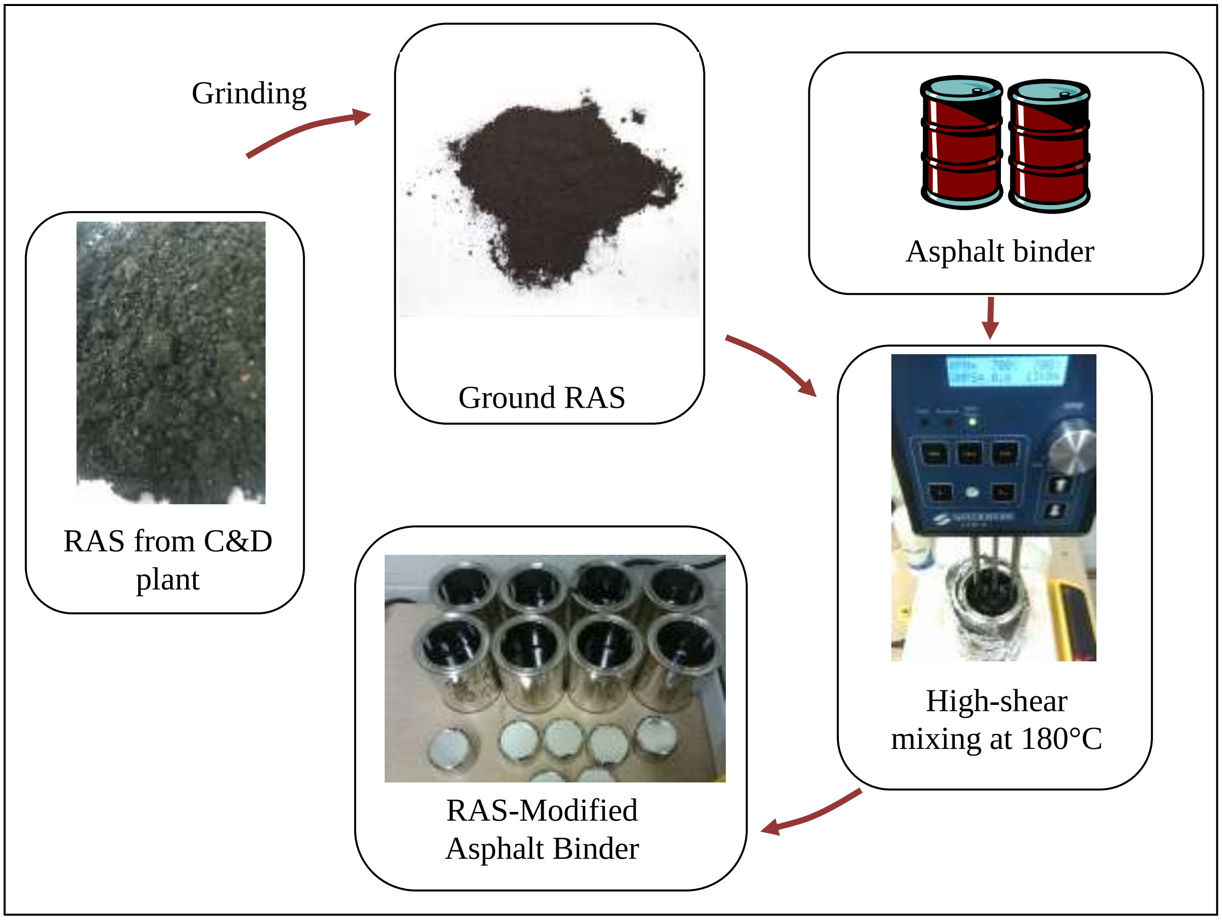


Figure 8. Preparation of RAS-Modified Asphalt Binder Using the Proposed Wet Process

Asphalt binder blends consisting of the unmodified binder and the ultra-fine RAS were prepared at modification proportions of 10, 20, and 40% by weight of the binder (Table 5). The blends were prepared by mixing 500 g of asphalt binder with the corresponding content of RAS at a mixing temperature of 180°C using a mechanical shear mixer rotating at a speed of 1500 rpm for 30 minutes as shown in figure 8. In addition to the prepared blends, a virgin air-blown binder commonly used in the manufacturing of shingles was tested (referred to as SHIN) (Table 5).

Table 6. Summary of Particle Size Analysis Using Laser Diffraction

RAS Type	Mean (µm)	Median (µm)	SD (µm)
Tear-off	99.5	60.3	119.0
Manufactured	201.0	133.0	196.0

3.3 Laboratory Testing

Laboratory testing activities in this study determined the effects of RAS modification on the binder rheological properties, molecular and fractional compositions, and compatibility of the blends when the wet process is used.

3.4 Superpave Binder Testing

Prepared blends were characterized using fundamental rheological tests (i.e., dynamic shear rheometry, rotational viscosity, and bending beam rheometer) and by comparing the Superpave Performance Grade (PG) of the RAS-modified blend to the unmodified binders as per AASHTO M 320-09 (Standard Specification for Performance-Graded Asphalt Binder).

3.5 Confocal Laser-Scanning Microscopy

Microscopic analysis of the microstructure of the prepared asphalt blends was conducted using Confocal Laser-Scanning Microscopy (CLSM) in a fluorescence mode. This method was selected given its ability to identify the broad fractions of asphalt binder including wax crystals and the simple sample preparation that does not affect the microscopic structure of the binder (Bearsley et al., 2004; Lu et al., 2005). When illuminated with a point laser source of a wavelength that causes fluorescence, wax crystals are detected in the binder as light-colored flecks. A Leica TCS SP2 microscope was irradiated with 488nm wavelength light and the fluorescence was observed in the range of 500-550 nm wavelengths. All images were captured as two-dimensional images in 1024 x 1024 bit TIFF format.

As suggested by Bearsley et al. (2004), microscopic samples were prepared by heating the asphalt blends to a fluid state, rigorously stirring the blend, and then pouring a small drop on a glass slide. To ensure a uniform and a thin depth of the sample, a cover slip was placed on top of the drop of the asphalt blend while still in a fluid state. The glass slide was then placed on a heated plate at 120°C and was left for 15 minutes until the drop would flow under the weight of the glass slip to cover the entire width of the slip.

3.6 Cigar Tube Test

The compatibility and stability of the prepared blends were evaluated using the cigar tube test (ASTM D 7173-05), which is used to determine the separation tendency of polymer-modified asphalt in the laboratory. In this test, 50 g of the prepared asphalt blends was poured in a sealed aluminum tube that was kept in a vertical position for 48 h at a temperature of 163 ± 5°C. At the end of the conditioning period, the top and bottom parts of the tube were separated and were tested using the Dynamic Shear Rheometer (DSR). Results were used to assess the stability and level of separation of the blends by calculating the percent separation (Jensen and Abdelrahman, 2006):

$$\text{Separation}= \frac{(G^*/\sin\delta)_{max}-(G^*/\sin\delta)_{avg}}{(G^*/\sin\delta)_{avg}}\text{x}100$$

Where;

G^* = complex shear modulus;

δ = phase angle;

$(G^*/sin\delta)_{max}$ = higher value of either the top or the bottom portion of the tube;

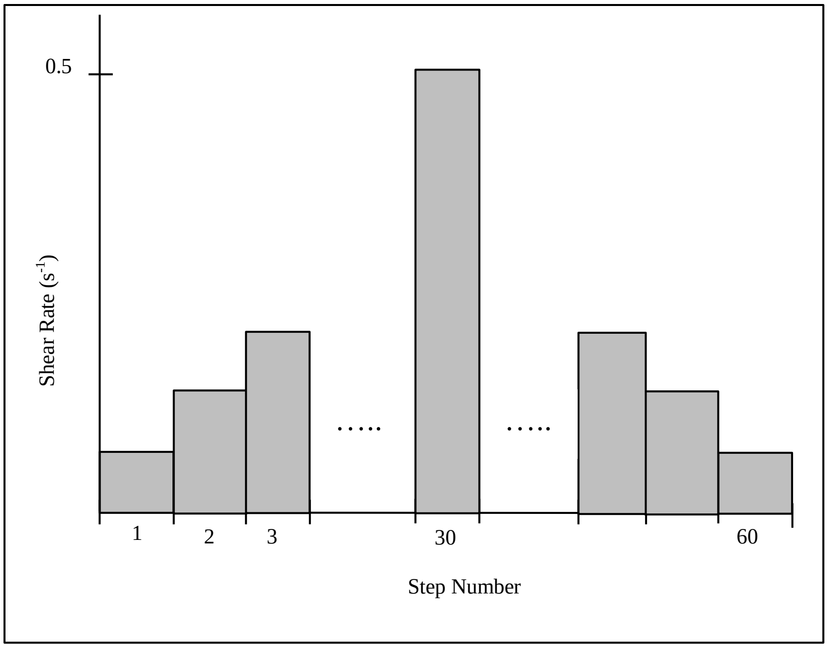
$(G^*/sin\delta)_{avg}$ = average value of the top and the bottom portions of the tube.

3.7 High Pressure Gel Permeation Chromatography (HP-GPC)

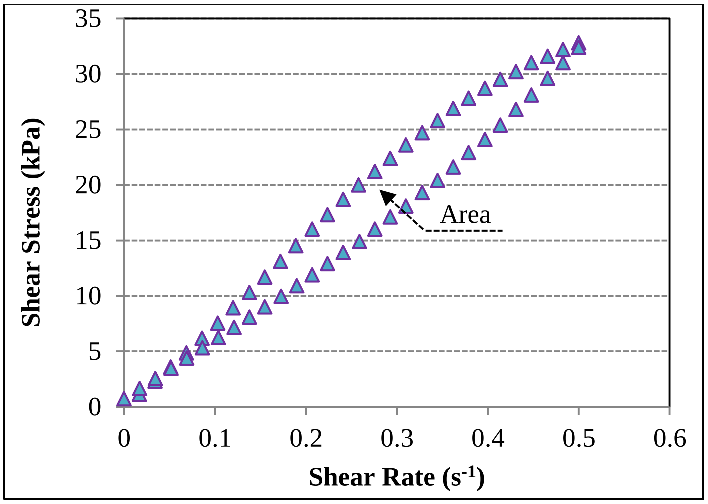
HP-GPC was conducted for a number of the prepared asphalt blends. A gel permeation chromatograph Agilent 1100 equipped with an auto injector and a Hitachi differential refractive index detector was used. The separation of the asphalt components was performed with three columns connected in series with pore sizes of 500 angstrom (Å), 10^{-4} Å, and mix beads. The column set was calibrated with narrow molecular weight polystyrene (PS) standards using 1wt% in tetrahydrofuran (THF). The elution volume observed for polystyrene standards with each given molecular weight was used to build a calibration curve. All asphalt samples for GPC were prepared at a concentration of 3 wt% in THF, injected through a 0.45µ filter into 150 µL vials, and inserted in an automatic sample injector. Samples were eluted with THF at 1 ml/min. at room temperature, and the species concentration in the eluent was recorded using a differential refractometer. The GPC curves were integrated, and the areas were normalized over the total area of the chromatogram. The expected error in the measured molecular fractions is around 0.2% or less. Two replicates were measured for each binder blend and the average was used in the analysis.

3.8 Thixotropy Testing

Three rheological test methods are used to study thixotropy of non-Newtonian fluids (Shu-Xin and Chuan-Jing 2006): thixotropy-loop experiment, which was adopted in this study, start-up experiment, and the oscillatory shear experiment. The hysteresis method, which consists of subjecting the asphalt specimen to a triangular loop; i.e., a linearly increasing shear rate followed by a linearly decreasing shear rate, was used, figure 9.a. If the binder exhibits thixotropic behavior, the increasing and decreasing curves do not coincide, causing a hysteresis loop. The area enclosed by the hysteresis loop under steady state conditions can be used as a measure of thixotropy, figure 9b. An Anton Paar MCR 302 rheometer with parallel plate configuration was operated on a triangular loop mode to conduct the thixotropy experiments at three test temperatures, 5, 25, and 50°C. Two replicate specimens were tested at each temperature for each binder blend. Sample geometry consisted of a 8-mm diameter and a 2-mm thickness at 5°C and 25°C and a 25-mm diameter and a 1-mm gap at 50°C. Each sample was tested for a total of 10 cycles, each with a maximum shear rate of 0.5 s^{-1} as recommended by previous research studies (Mewis 1979, Mouillet 2012).



(a)



(b)

Figure 9. (a) Description of the Hysteresis Test Procedure and (b) Typical Hysteresis Loop from the Thixotropy Experiment

3.9 Viscosity Testing

The temperature susceptibility of the base binder and RAS-modified asphalt blends was evaluated by developing temperature-viscosity plots for the prepared samples. In addition to the blends presented in table 5, viscosity testing was also conducted at a RAS content of 30% to evaluate the effects of RAS on the binder viscosity at high content. Three replicates were tested for each prepared blend and base binder with an average coefficient of variability (COV) of 5%. A Brookfield rotational viscometer was used at a test temperature ranging from 95 to 175°C according to the procedure outlined by ASTM D 4402. While the experimental program intended to measure viscosity starting from 50°C, the flow of the blends was not sufficient at this temperature to ensure accurate measurements of the viscosity. The Viscosity-Temperature Susceptibility (VTS) was calculated for the temperature range between 95 and 135°C based on Equation below (Roberts et al. 1996):

$$\mathrm{VTS} = \frac{\mathrm{loglog}(\eta_1) - \mathrm{loglog}(\eta_2)}{\log(T_2 + 273.15) - \log(T_1 + 273.15)}$$

where,

η_1 and T_1= viscosity in Pa.s at T_1 = 95°C; and

η_2 and T_2= viscosity in Pa.s at T_2 = 135°C.

3.10 Dynamic Mechanical Analysis

Dynamical mechanical analysis (DMA) was performed using an Anton Paar MCR 302 rheometer with parallel plate configuration in a strain-controlled mode. The test temperatures ranged from 5 to 75°C in increments of 10°C. Frequency sweeps were performed on all samples over the entire range of temperatures. Twenty frequencies were used at each test temperature ranging from 0.1 to 100 Hz. This wide range of frequencies allowed a strong overlap between the test temperatures in the construction of master curves. The main results of the dynamic mechanical analysis were the complex shear moduli (G*) and phase angles (δ) over the entire range of applied frequencies at each tested temperature.

In order to analyze the effects of RAS modification on the behavior of the binder at intermediate and high service temperatures, the dynamic mechanical functions obtained from the DSR were shifted in the form of master curves of G* and δ. The response curve at 25°C was considered the reference temperature, and all isothermal segments were shifted along the frequency axis to obtain a smooth master curve. Isochronal plots of complex modulus and phase angle were also developed at a frequency of 0.1, 11.3, and 100 Hz and were used to investigate the effects of RAS modification on the binder rheological properties.

CHAPTER 4: RESULTS AND ANALYSIS

The results obtained from rheological and molecular tests of asphalt binder are presented and discussed in this chapter. The analysis of experimental results allowed to assess the effectiveness of RAS on asphalt binders.

4.1 Superpave Binder Testing

Tables 7 and 8 present the measured rheological properties of the RAS-modified and unmodified binders as well as their final PG grades based on laboratory testing conducted using rotational viscometer, dynamic shear rheometer, and bending beam rheometer. Results are presented for 13 types of blends: PG 64-22 conventional, PG 64-22 + 10, 20% manufactured, + 10, 20% tear-off, and the pure shingle binder (SHIN); and PG 52-28 conventional, PG 52-28 + 10, 20, 40% manufactured, + 10, 20, 40% tear-off. As shown in tables 7 and 8, the use of RAS as a modifier to the binder increased its viscosity, stiffened the binder at high temperature, and reduced its elongation properties at low temperature. This was expected as the binder used in shingle manufacturing and present in RAS materials is an air-blown asphalt binder with stiff characteristics and low elongation properties. In fact, the shingle binder (SHIN) was ranked by the Superpave binder specification system as PG 100 and did not pass the m-value criterion at low temperature even when tested at 0°C (all values were below passing limit). These results indicate that the use of RAS modification would generally improve or not influence the high temperature grade of the binder but it may reduce elongation characteristics of the binder at low temperature. However, given that the requirements change when the high temperature grade is shifted (e.g., from 52 to 58°C), an optimum shingle content may be identified that will improve the high temperature grade without influencing the low temperature grade of the binder (e.g., 52T20 and 52M20 with a final PG grade of 58-28). These results highlight the benefits of the proposed wet process in controlling the final PG grade of the binder when RAS material is used. The final PG grade of the binder when RAS is incorporated into the mix cannot be directly measured.

Table 7. Results of the Superpave PG Testing (PG 64-22)

Binder Testing	Spec	Test Temp	PG 64-22	64M10	64M20	64T10	64T20	SHIN
Test on Original Binder								
Dynamic Shear, G*/Sin(δ), (kPa), AASHTO T315	1.00^{+}	64°C	2.16	2.66	2.7	3.06	4.165	1.08 (100°C)[1]
	1.00^{+}	70°C	0.993	1.28	1.23	1.38	1.91	----
Rotational Viscosity (Pa·s), AASHTO T316	3.0^{-}	135°C	0.48	0.53	0.67	0.69	0.70	3.74
Tests on RTFO								
Dynamic Shear, G*/Sin(δ), (kPa), AASHTO T315	2.20^{+}	64°C	4.37	5.15	7.07	11.2	7.42	2.49 (100°C)[2]
	2.20^{+}	70°C	1.96	2.29	3.11	4.08	3.35	----
Tests on (RTFO+ PAV)								
Dynamic Shear, G*Sin(δ), (kPa), AASHTO T315	5000^{-}	28°C	2940	4050	3910	3925	3350	4185 (25°C)[3] 5185 (22°C)[4]
BBR Creep Stiffness, (MPa), AASHTO T313	300^{-}	-6°C	88	90	108	89	111	43 (0°C)
		-12°C	189	209	227	179	195	66 (-6°)
Bending Beam m-value AASHTO T313	0.300^{+}	-6°C	0.364	0.356	0.332	0.344	0.365	0.290 (0°C)[5]
		-12°C	0.322	0.285	0.287	0.278	0.298	0.261 (-6°C)[6]
Actual PG Grading			**PG 64-22**	**PG 70-16**	**PG 70-16**	**PG 70-16**	**PG 70-16**	**PG 100**

1 and 2 The binder passed all temperatures, so it was tested at 100˚C
3 and 4 The binder needs to be tested at different temperature because of different low temperature criteria
5 and 6 The binder didn't pass low temperatures and need to be tested at higher temperatures.

Table 8. Results of the Superpave PG Testing (PG 52-28)

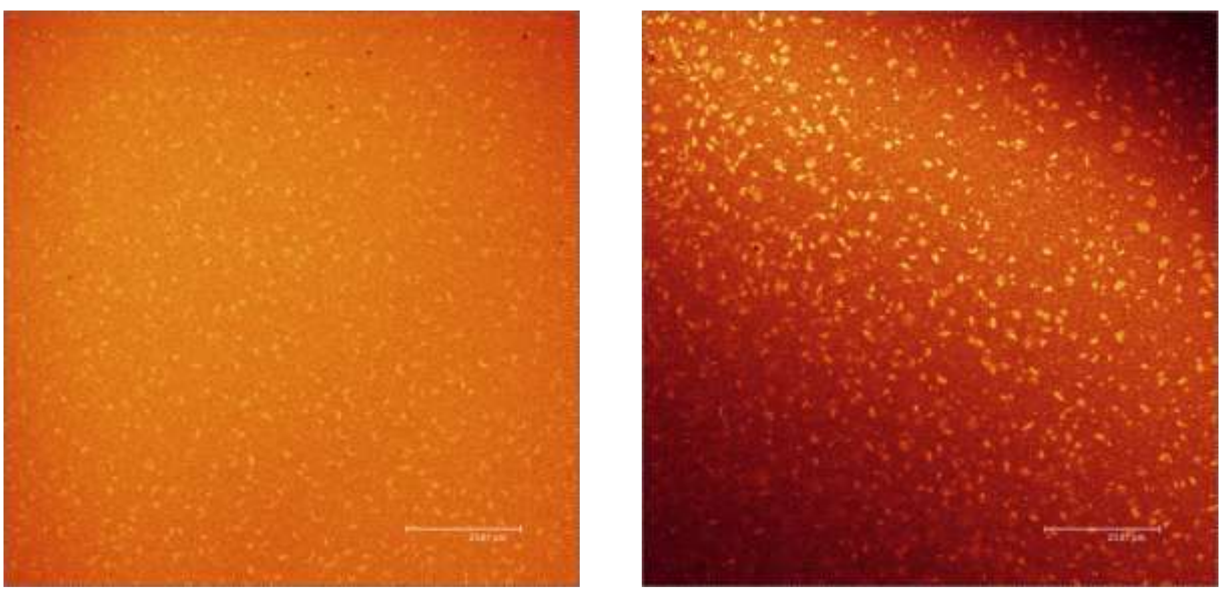
Binder Testing	**Spec**	**Test Temp**	**PG 52-28**	**52M10**	**52M20**	**52M40**	**52T10**	**52T20**	**52T40**
		Test on Original Binder							
Dynamic Shear, G*/Sin(δ), (kPa), AASHTO T315	1.00^{+}	58°C	1.02	1.08	1.29	2.48	1.07	1.52	3.49
Rotational Viscosity (Pa·s), AASHTO T316	3.0^{-}	135°C	0.213	0.233	0.296	0.444	0.238	0.306	0.341
		Tests on RTFO							
Dynamic Shear, G*/Sin(δ), (kPa), AASHTO T315	2.20^{+}	52°C	4.07	4.59	5.82	----	4.33	7.44	----
		58°C	1.81	1.98	2.43	3.84	1.94	3.08	3.86
		Tests on (RTFO+ PAV)							
Dynamic Shear, G*Sin(δ), (kPa), AASHTO T315	**5000^{-}**	16°C	4920	5345	5595	5020 (22°C)[1]	6070	6150	4780 (22°C)[2]
		19°C	3135	3380	3585	3295 (25°C)[3]	3870	4030	3150 (25°C)[4]
BBR Creep Stiffness, (MPa), AASHTO T313	300^{-}	-12°C	91	82	107	146	86	115	135
		-18°C	227	224	259	313	255	256	473
Bending Beam m-value AASHTO T313	0.300^{+}	-12°C	0.405	0.394	0.382	0.347	0.383	0.379	0.341
		-18°C	0.330	0.325	0.322	0.298	0.324	0.319	0.280
Actual PG Grading			**PG 52-28**	**PG 52-22**	**PG 58-28**	**PG 58-16**	**PG 52-22**	**PG 58-28**	**PG 58-22**

1, 2, 3 and 4 The binder needs to be tested at different temperature because of different low temperature criteria

4.1 Confocal Laser-Scanning Microscopy

Prepared microscopic samples were visualized using CLSM to reveal the concentration of wax crystals in the pure binder and the prepared blends as well as the effects of RAS on the microscopic features of the binder. Figure 10 presents a comparison between the images of PG 52-28 pure binder and the air-blown asphalt binder (SHIN) used in the manufacturing of shingles. As shown in these images, a continuous phase is observed, in which the wax crystals are dispersed and are manifested as light-colored particles. These molecules were reported to have between 20 to 40 carbons and a melting temperature between 60 and 90°C (Lu et al., 2005; Lesueur, 2009).

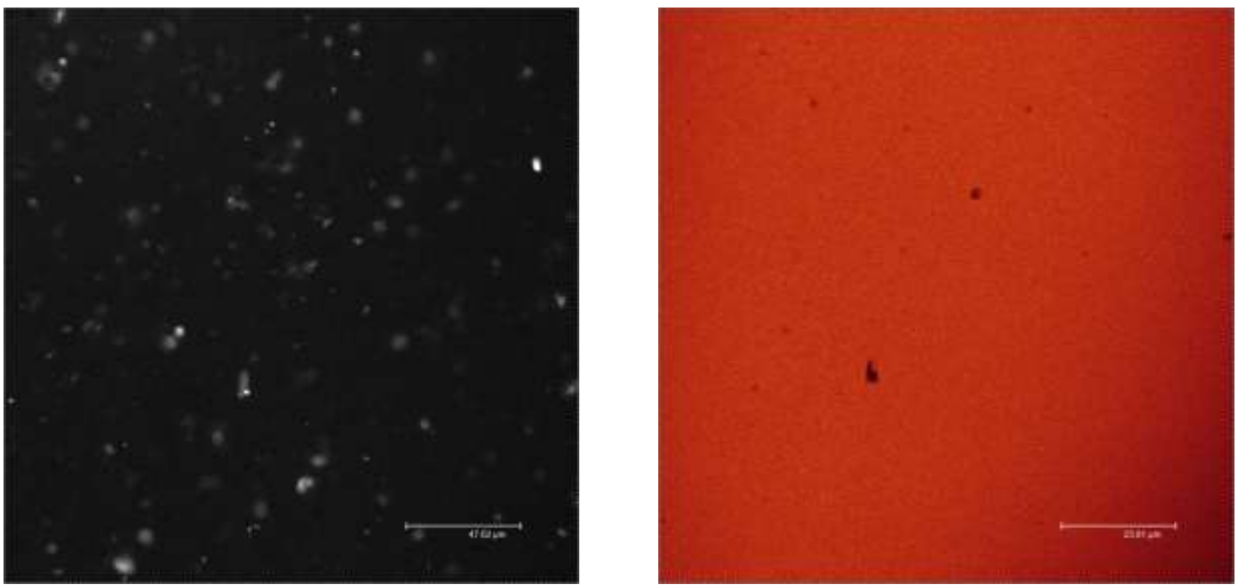
The size of the wax particles ranged from 4 to 8 microns with a flake shape, which is in agreement with the findings of past research (Lu et al., 2005). The size and concentration of wax crystals were greater in the air-blown asphalt binder than in the PG 52-28 binder, figure 10. The concentration and morphology of wax particles is believed to have an impact on the binder performance (Lu et al., 2005). Therefore, the greater concentration of wax crystals in the air-blown asphalt may cause this binder to be stiffer and more brittle than the soft PG 52-28 binder, which showed lower concentration of wax molecules.



(a) 52-28 (b) SHIN

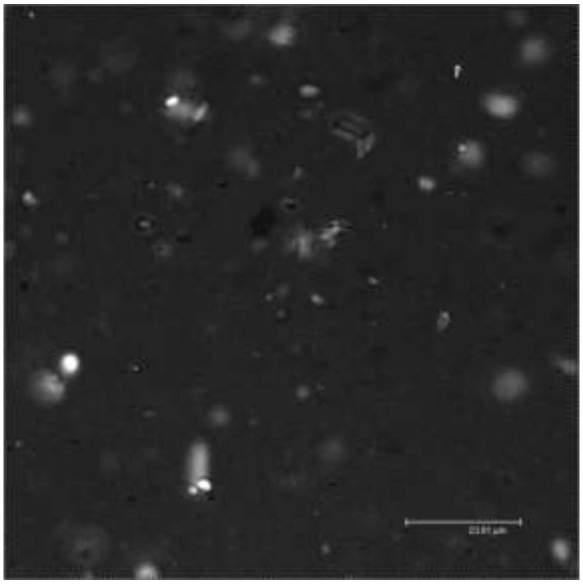
Figure 10. CLSM Images of Control Binder (52-28) and Pure Air-Blown Asphalt Binder

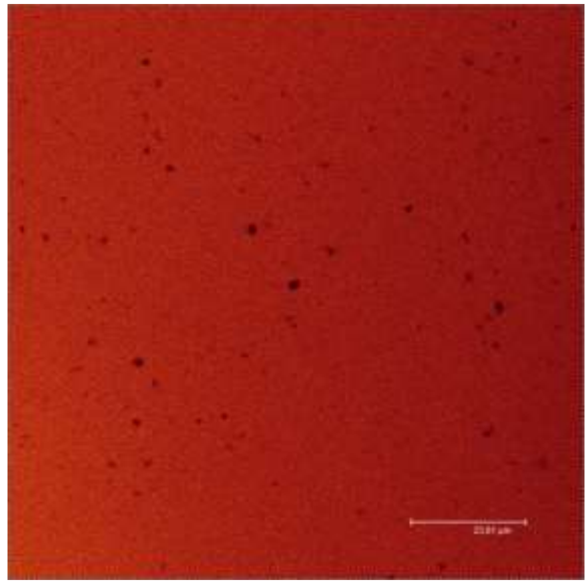
Fig. 11 presents the optical and fluorescence microscopic images of the blend prepared with PG 52-28 + 20% ground RAS shingles from Maine (52M20). In these figures, the ground mineral particles are observed in the optical images and are dispersed in the asphalt phase. However, the fluorescence microscopic images do not show the wax crystals as it was observed in the pure binder. In fact, the components of the blend did not fluorescence in the CLSM images. The absence of fluorescence in the images of the blend may be due that the wax crystals are absorbed by the RAS material and create a new phase that is not fluorescent. The same trend was observed for the blend prepared with PG 52-28 + 40% ground RAS shingles from Missouri (52T40), Figure 12. As shown in this figure, the absence of fluorescence particles may indicate that the wax crystals were absorbed by the RAS binder.



(a) 52M20 – Optical (b) 52M20 - Fluorescence

Figure 11. CLSM Images of RAS-Modified Binder Prepared with 20% Ground Shingle





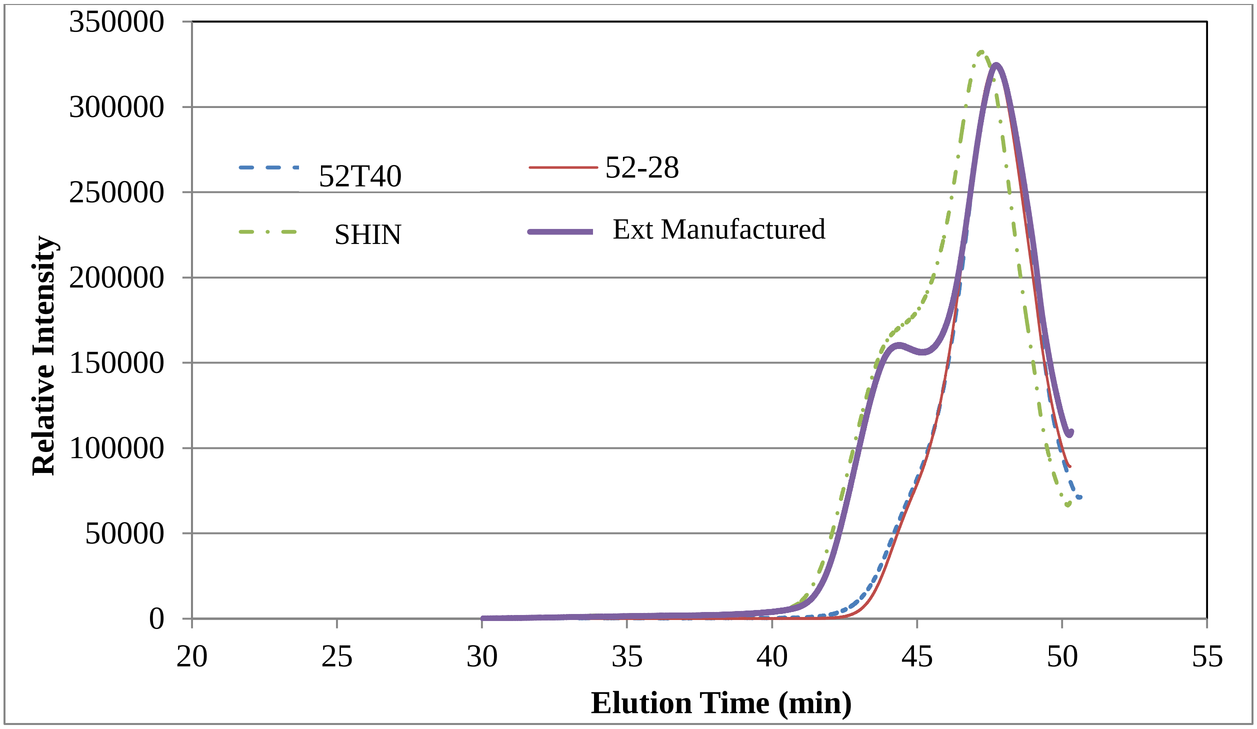
(a) 52T40 – Optical

(b) 52T40 - Fluorescence

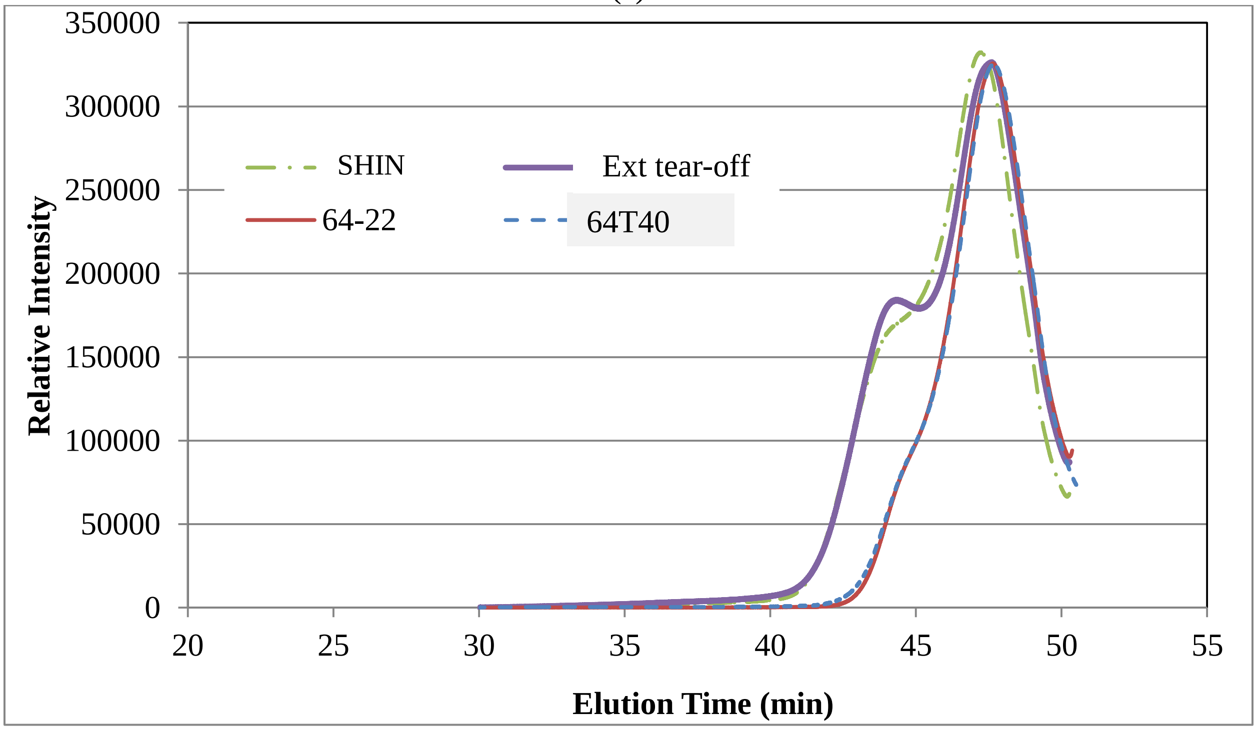
Figure 12. CLSM Images of RAS-Modified Binder Prepared with 40% Ground Shingle

4.2 High-Pressure Gel Permeation Chromatography Analysis

Figure 13 (a and b) presents the HP-GPC chromatograms for the control binders, the prepared blends using RAS, the air-blown asphalt binder (SHIN) used in the manufacturing of shingles, and the extracted binder for the RAS shingles from Missouri (EXT Tear-off) and from Maine (EXT manufactured). As shown in this figure, the addition of RAS resulted in a slight shift in the molecular side distribution (MSD). This shift was not significant as the 0.45µ filter used in the experiment retained the majority of the fillers in the RAS materials. The significant shift in the chromatogram of the extracted binder and the air-blown asphalt to the left is indicative that the high molecular weight (HMW) fraction in these binders is greater than in the soft PG 52-28 and the regular PG 64-22 binders.



(a)

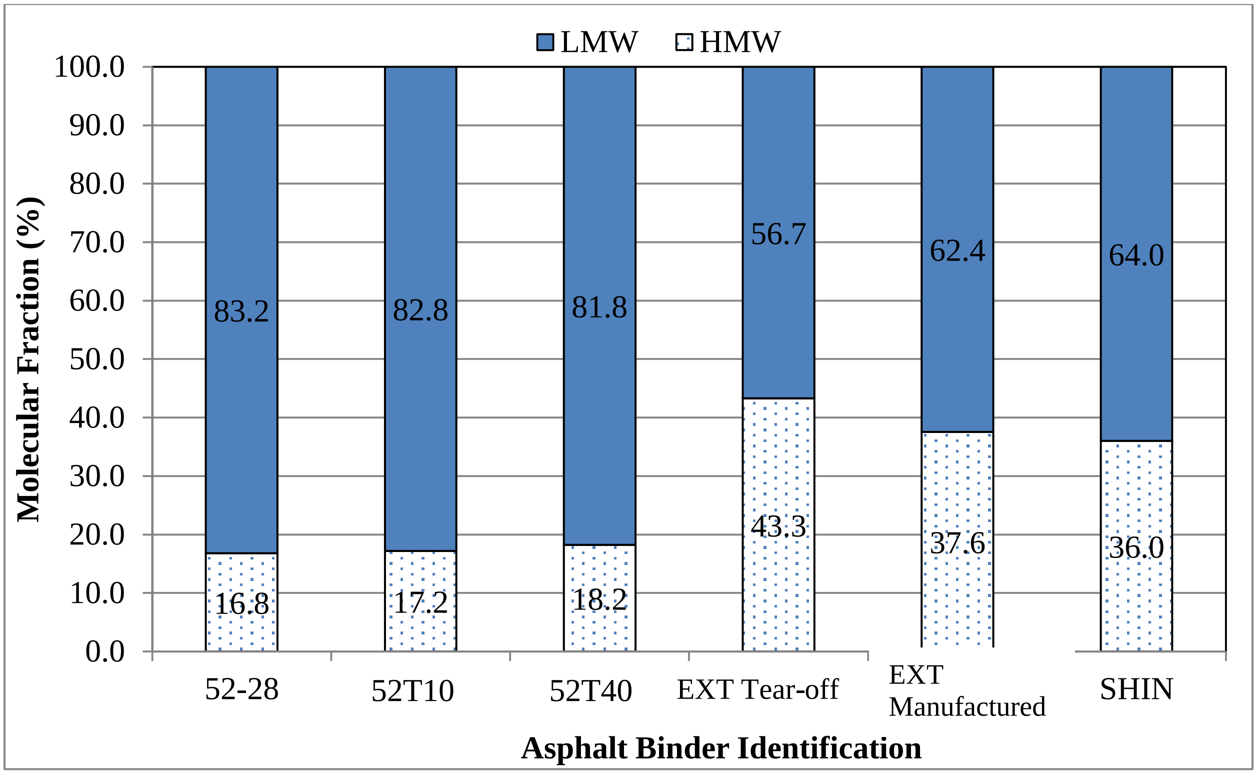


(b)

Figure 13. HP-GPC Chromatograms for Base, RAS-Modified, and Shingle Binders

Figure 14 (a and b) presents the molecular size distribution obtained from the HP-GPC test results for the control binders, the prepared blends using RAS, the air-blown asphalt binder

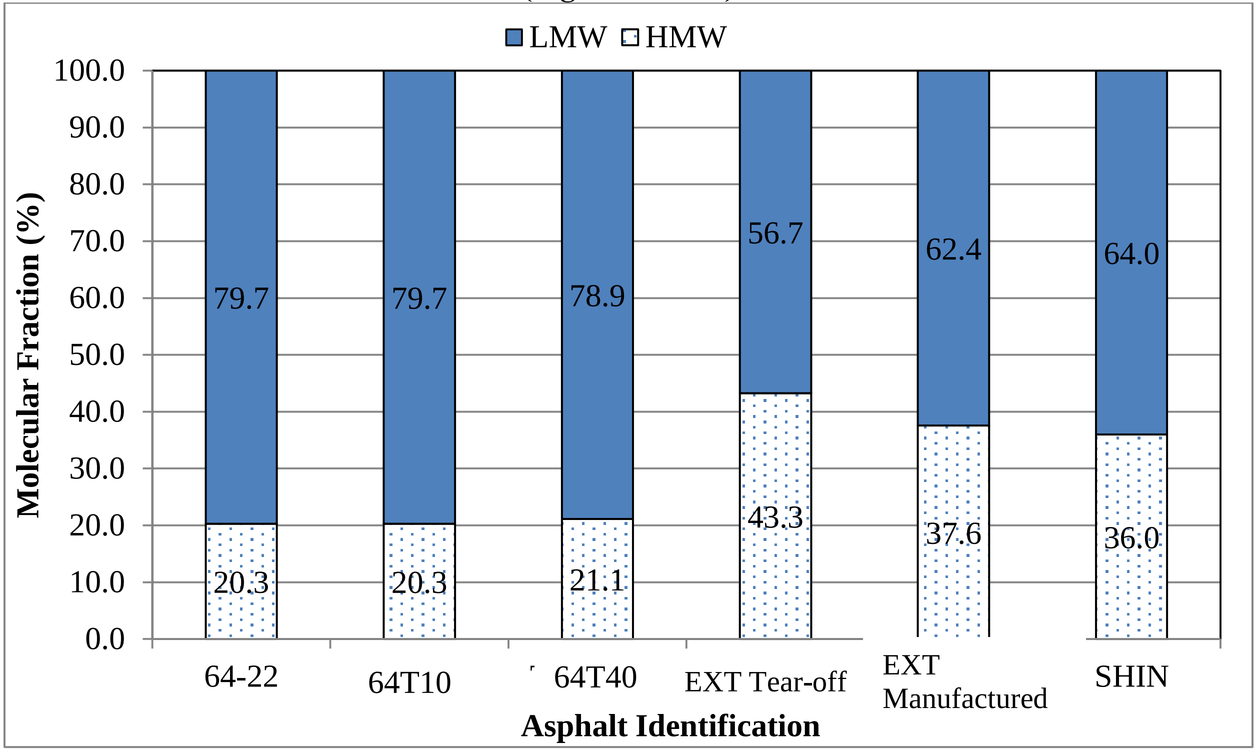
(SHIN) used in the manufacturing of shingles, and the extracted binder for the RAS shingles from Missouri (EXT tear-off) and from Maine (EXT manufactured). Fractional composition of the binders were divided into two main groups: (1) high molecular weight (HMW), which represents the molecular fraction in the binder with a molecular weight of 3,000 or greater and (2) low molecular weight (LMW), which represents the molecular fraction in the binder with a molecular weight of 3,000 or smaller. Past research conducted at LSU concluded that an increase in the binder content of LMW results in an increase in its elongation properties at intermediate and low temperatures (Elseifi et al. 2010).



(a)

Figure 14. Molecular Fractional Distributions for Unmodified Binders and RAS-Modified Binders for (a) PG 52-28 and (b) PG 64-22

(Figure cont'd.)

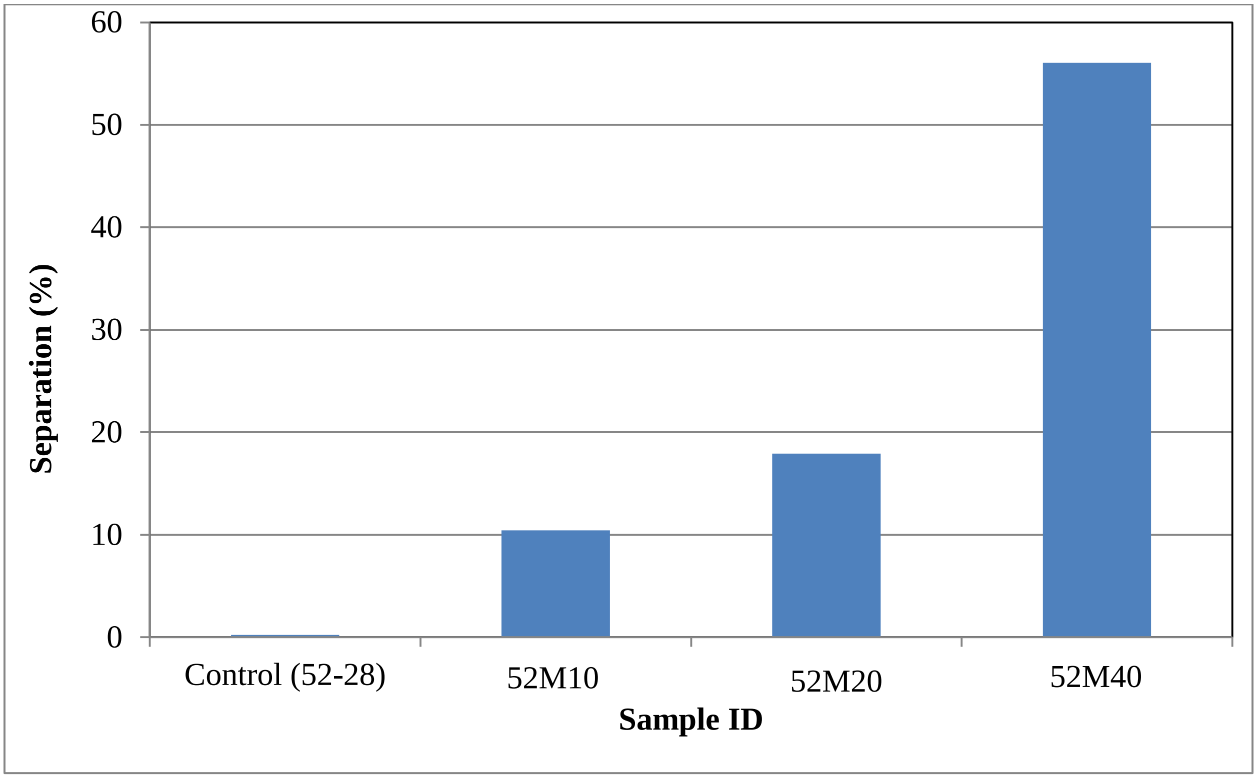


(b)

Results presented in figure 14 (a and b) identify the effects of RAS modification on the binder molecular compositions. As shown in figure 14, the extracted binder from RAS (i.e., EXT tear-off and EXT manufactured) had a high content of HMW, which was expected as this binder is manufactured for such high content. The proposed wet method of modification caused a slight increase of the HMW content in the prepared blends especially at high content of RAS modification (PG 52-28 vs. 52T40 and 64-22 vs. 64T40). The increase in HMW was not significant at low content of modification as the major part of the ground RAS is composed of mineral fiber and mineral and ceramic-coated granules with only about 20% of binder.

4.3 Cigar-Tube Test

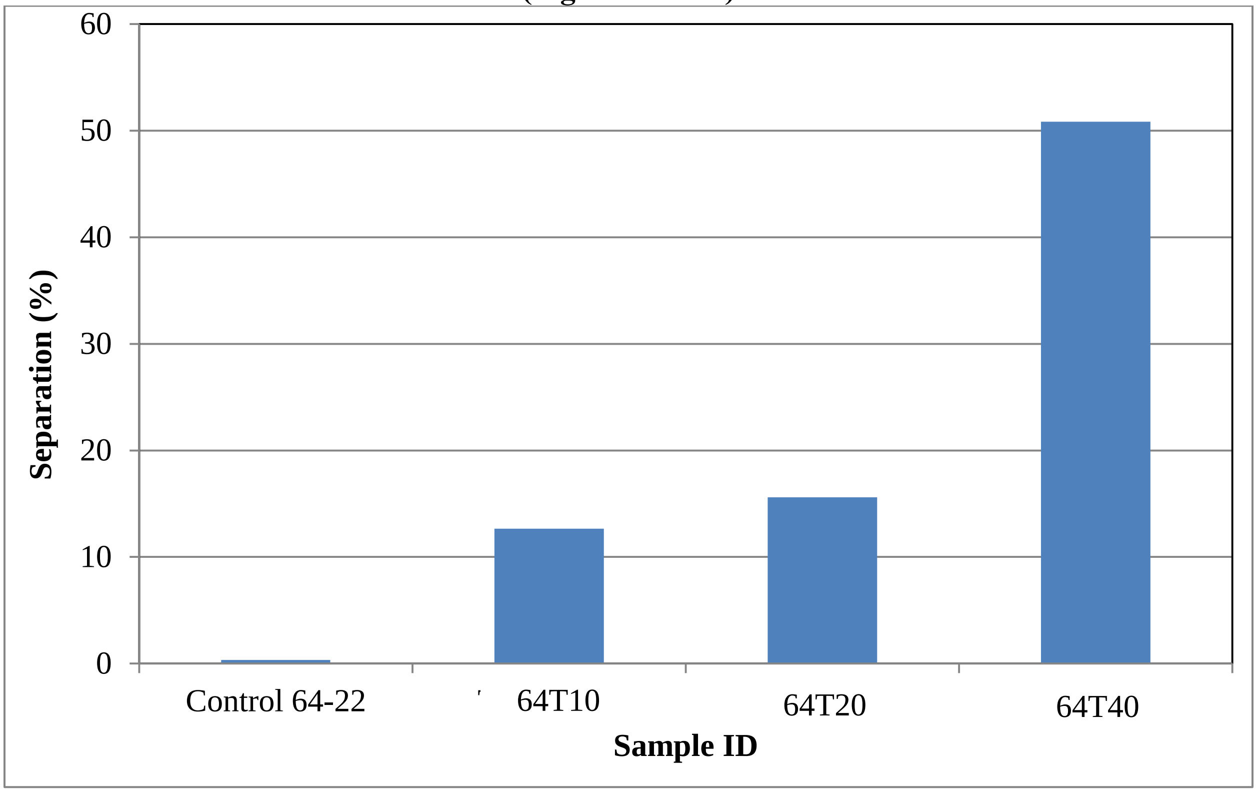
Figure 15 (a and b) presents the results of the cigar tube test for the unmodified binders and the prepared blends using RAS. For Crumb Rubber Modified (CRM) binder, a level of separation of 10 to 15% or less is recommended (Jensen and Abdelrahman, 2006). Levels of separation were calculated according to Equation (1) using DSR test results. As shown in figure 15, the use of a RAS content of 20% or less resulted in levels of separation less than 20%. At high RAS content of 40%, stability and workability of the blend will not be favorable given the high level of separation. This is due to the mineral fillers in the RAS material that settles after being held for 48 hours in a vertical position. To minimize separation during storage, a digestion tank equipped with an agitator and a super-heater should be used during production of shingle-modified asphalt based on the proposed wet process.



(a)

Figure 15. Levels of Separation in the Cigar Tube Test for Unmodified Binders and RAS-Modified Binders for (a) PG 52-28 and (b) PG 64-22

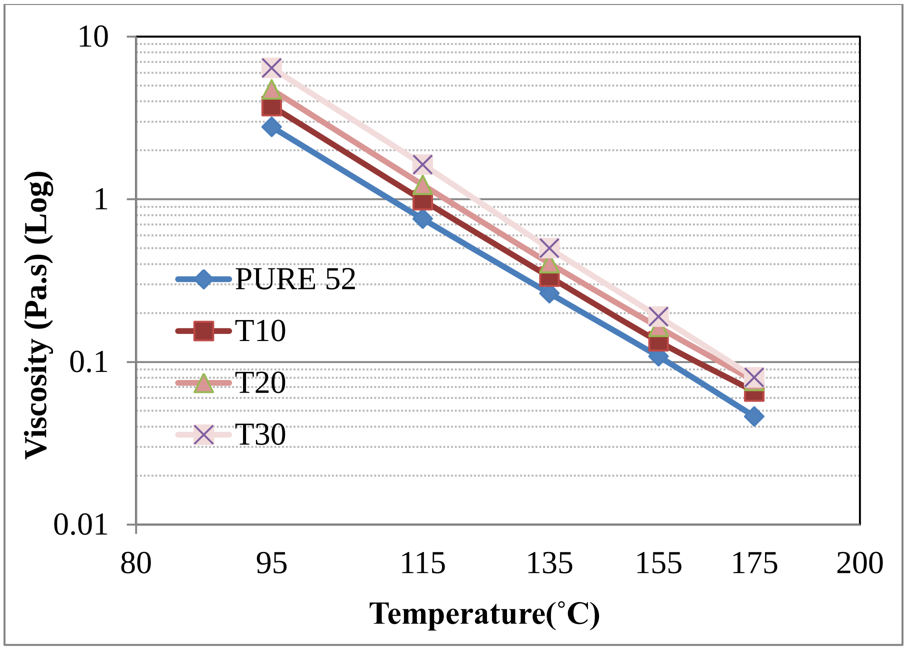
(Figure cont'd.)



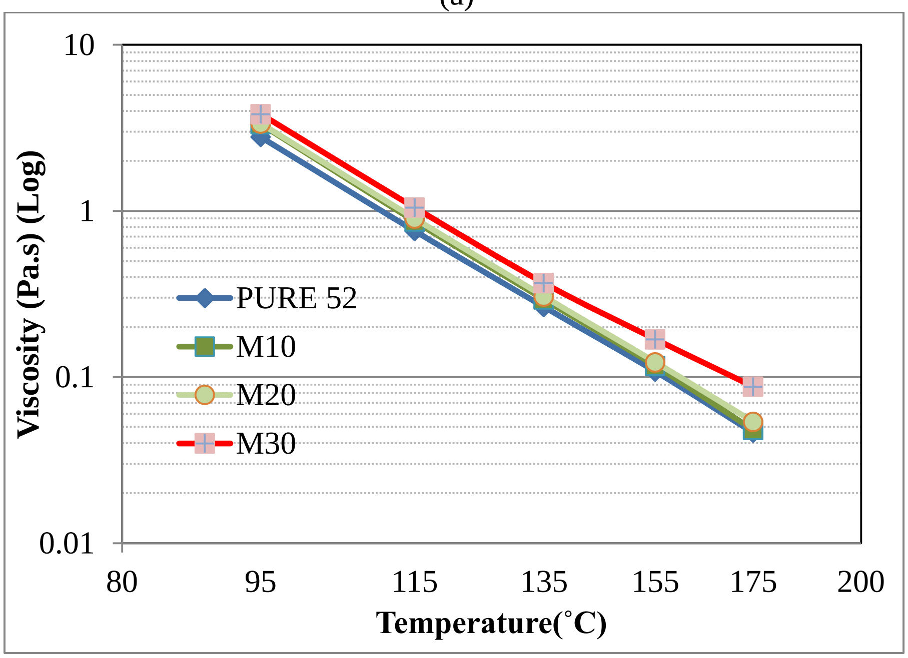
(b)

4.4 Viscosity Measurements and Temperature Susceptibility

Figures 16 (a and b) present the variation of the viscosity with temperature for the base binder and RAS-modified blends for tear off and scrap shingles from manufacturer waste, respectively. As shown in these figures, the relationship between viscosity and temperature is linear in a double logarithmic scale, which is in agreement with previous studies (Davis 2009). The use of RAS from tear-off shingles resulted in an increase in viscosity ranging from 20 to 130% when compared to the base binder. The increase in viscosity was proportional to the RAS content with greater increase at a RAS content of 30%. The use of RAS from manufacturer waste also resulted in an increase in viscosity ranging from 3 to 90% when compared to the base binder. However, the increase in viscosity for the blends prepared with RAS from tear-off was greater than for the blends prepared with RAS from manufacturer waste. This was expected as the asphalt binder in RAS from tear-off would age and lose light components during service.



(a)



(b)

Figure 16. Effects of RAS Modifications on the Viscosity-Temperature Relationships for (a) the Blends Prepared with RAS from Tear-Off Shingles and (b) the Blends Prepared with RAS from Scrap Shingles from Manufacturer Waste

Figure 17 presents the effects of RAS modifications on temperature susceptibility as expressed by the VTS. Larger VTS values indicate greater temperature susceptibility. As shown in this figure, the use of RAS did not considerably influence the VTS of the binder. For the blends prepared with RAS from tear-off, the temperature susceptibility of the binder in the range from

95 to 135°C decreased with the use of RAS. This may be due to the granules present in the RAS and that are typically temperature inert. For the blends prepared with RAS from manufacturer waste, temperature susceptibility of the binder increased at a RAS content of 10% but it then decreased at a RAS content of 30%. However, in both cases, the change in the binder VTS with the use of RAS was minimal.

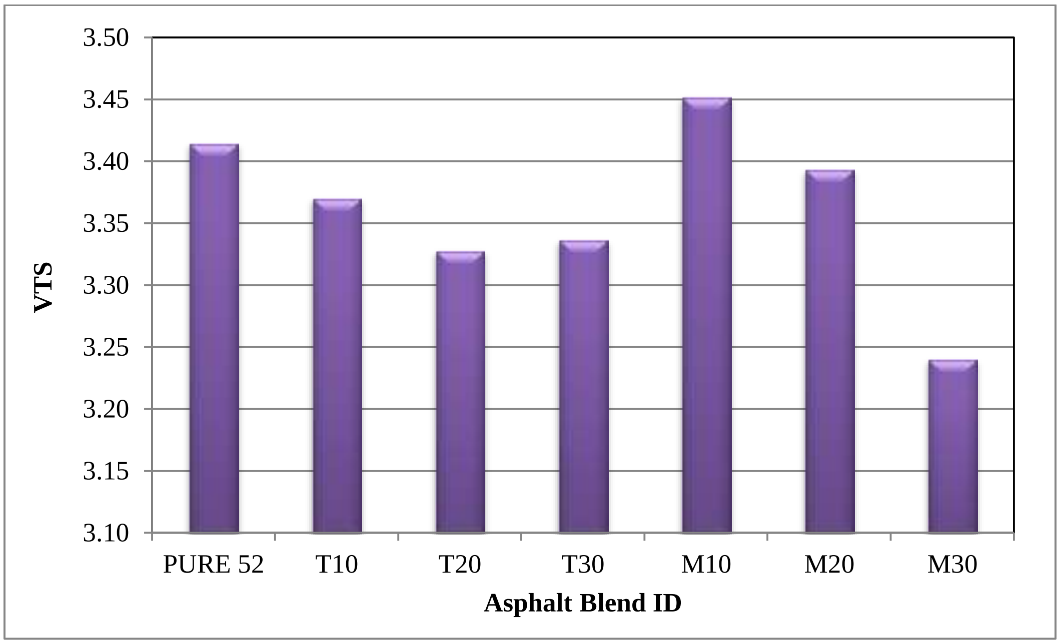
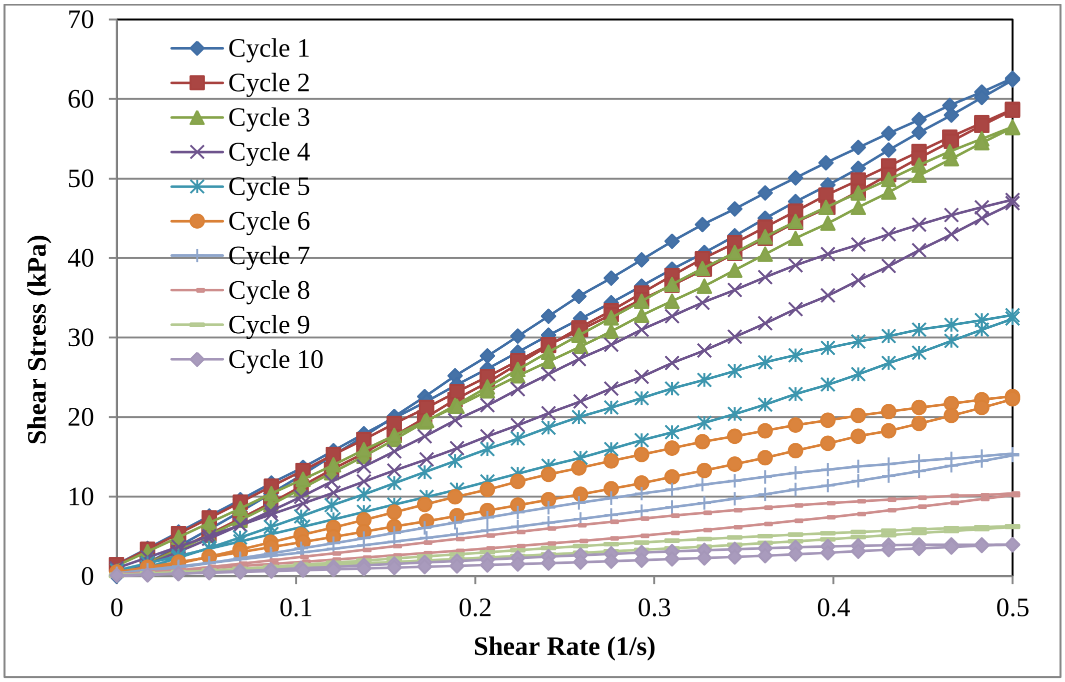


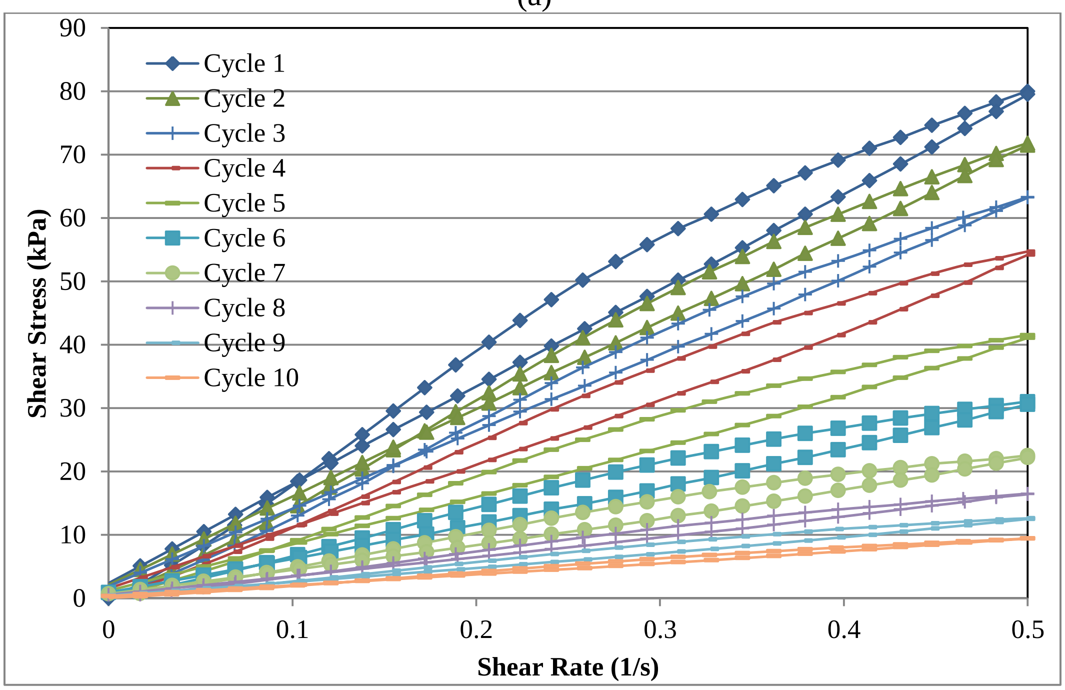
Figure 17. Effects of RAS Modification on the Viscosity-Temperature Susceptibility (VTS)

4.5 Thixotropy Testing

Figure 18 (a and b) presents the 10 hysteresis loops for the conventional PG 52-28 binder and the asphalt blend prepared with 20% RAS from tear-off (T20) at 25°C. As shown in this figure, loops continue to change and the hysteresis loops do not reach an equilibrium state, which is generally sought to eliminate the effect of shear history and to ensure that thixotropy is not confounded with other material characteristics such as viscoelastic relaxation (Mewis 1979). To investigate this effect, the variation of the apparent viscosity was plotted versus shear rate for each hysteresis cycle in the ascending shear step, figure 19. As shown in this figure, the viscosity of the binder slightly increased but then remained mostly constant over each cycle. However, a clear downward trend in viscosity was observed between each consecutive cycle, which is indicative of shear thinning. Shear thinning and thixotropy are often observed concurrently in colloidal dispersions (Mewis 1979). In this case, thixotropy is attributed to the breakage of chemical and mechanical bonds and its time-dependent recovery during rest. With cyclic repetitions, shear thinning results in a reduction of viscosity, which is observed through a gradual decrease in the shear stress carried by the material.



(a)



(b)

Figure 18. Hysteresis Loops for (a) Control 52-28 and (b) Asphalt Blend with 20% RAS from Tear-Off

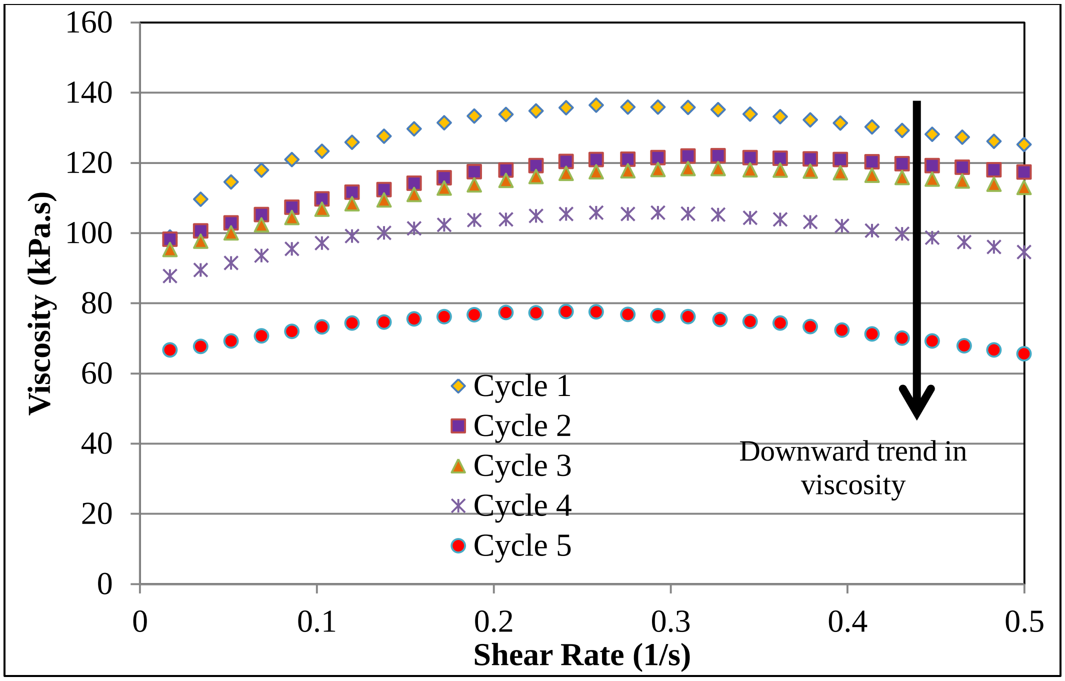
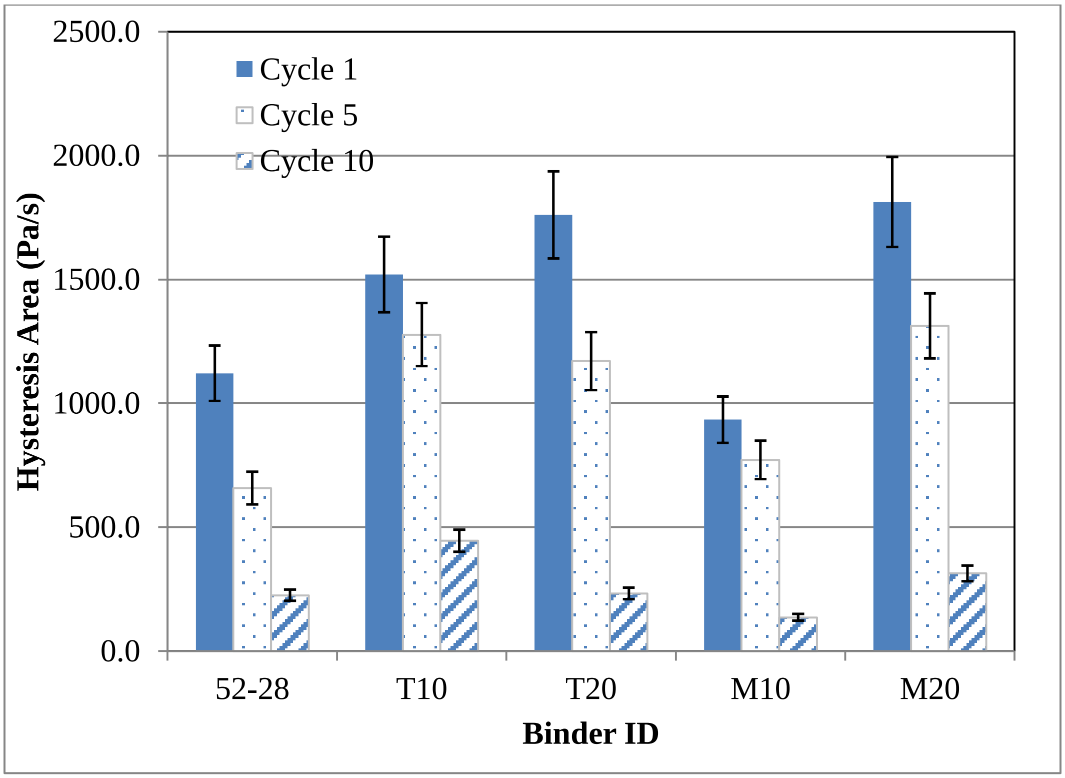


Figure 19. Variation of Viscosity with Cyclic Loading for Control PG 52-28 Binder

To relatively compare the thixotropic behavior of the different asphalt blends, the area enclosed in a loop after each cycle was calculated as an indicator of the level of thixotropy in each binder blend. Larger areas are indicative of greater susceptibility to thixotropy, which suggests that the binder is more sensitive to destructuring of the chemical and mechanical bonds but with a faster recovery during rest periods. Mouillet et al. reported that a binder, which was more susceptible to thixotropy but with a faster restructuring during recovery, had a superior mix fatigue performance (Mouillet et al. 2012). Figure 20a presents the calculated hysteresis areas enclosed in the first, fifth, and tenth cycles for the control and RAS-modified asphalt binders. Error bars showing an average variability of ±10% are shown in this figure, which is indicative of the average variability in the measurements. As shown in this figure, the RAS-modified binders exhibited greater hysteresis areas and more thixotropy than the control binder with the exception of the binder with 10% RAS from manufacturer waste (M10). In addition, thixotropy increased with the increase in RAS content for both tear-off and manufacturer waste. In the 10th cycle, all binders showed negligible hysteresis areas, which is indicative that thixotropy decreased with cyclic loading.

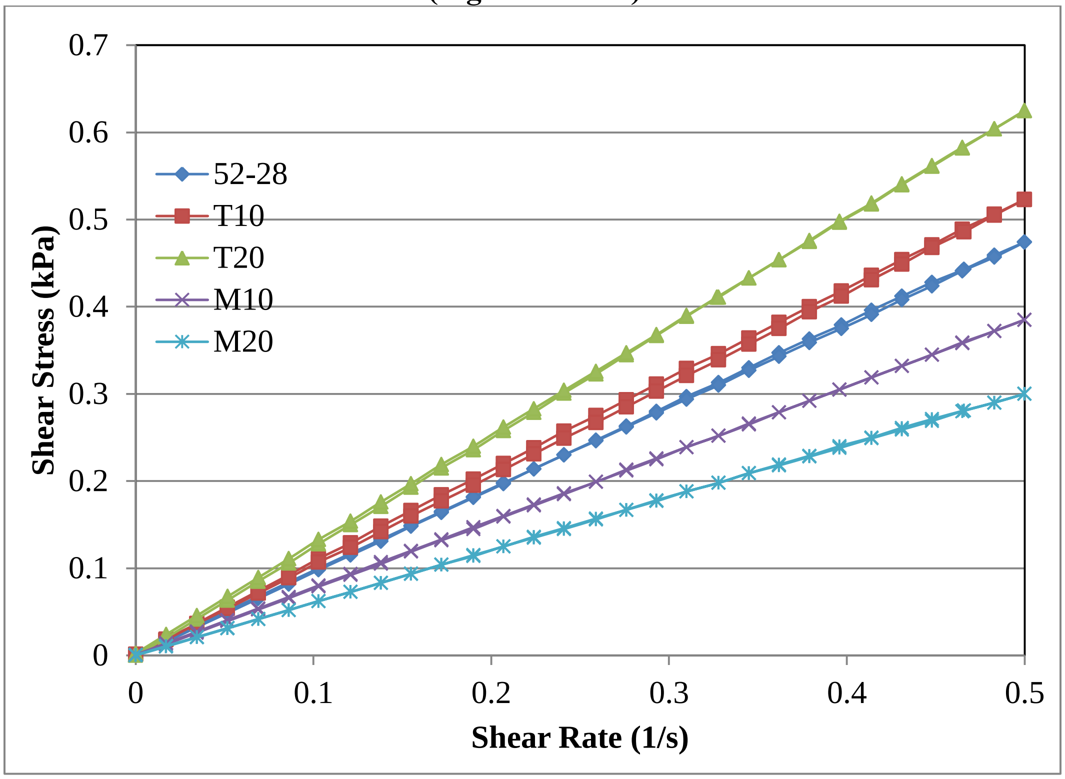
Figure 20b presents the hysteresis loops for the different asphalt binder blends at 50°C. As shown in this figure, the areas enclosed in the hysteresis loops are almost non-existent indicating that the thixotropy effects are negligible at high temperature. Similar trends were observed in the subsequent cycles with a downward trend in the applied shear stress with cyclic loading, which is indicative of a reduction in viscosity. It is also noted that the shear stress increased with the increase in RAS content for both tear-off and manufacturer waste, which is due to the stiffening effect of RAS. As noted in viscosity measurements, this effect was more pronounced for the blends prepared with RAS from tear-off than for the blends prepared with RAS from manufacturer waste.



(a)

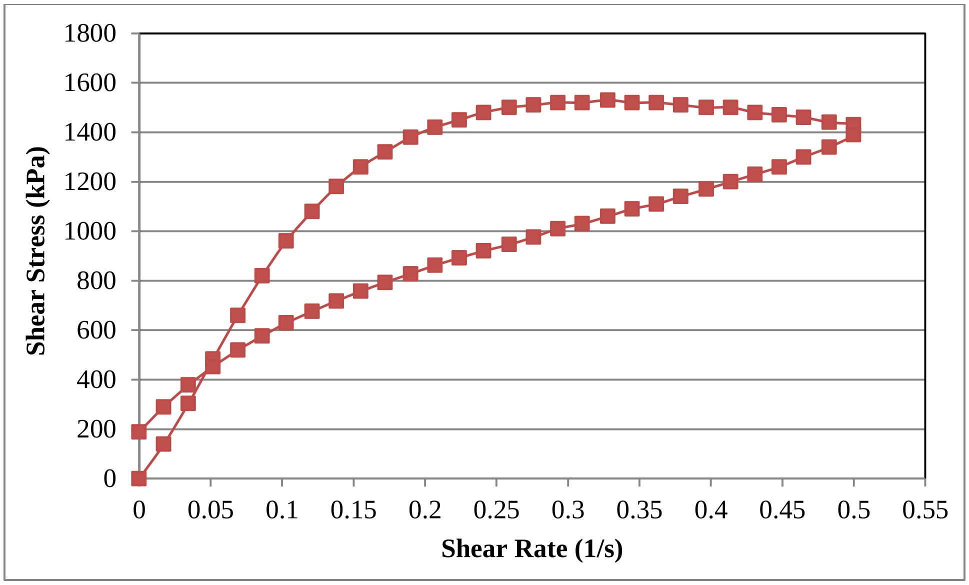
Figure 20. Hysteresis Analysis for Control 52-28 and RAS-Modified Asphalt Binders at (a) 25°C and (b) 50°C

(Figure cont'd.)

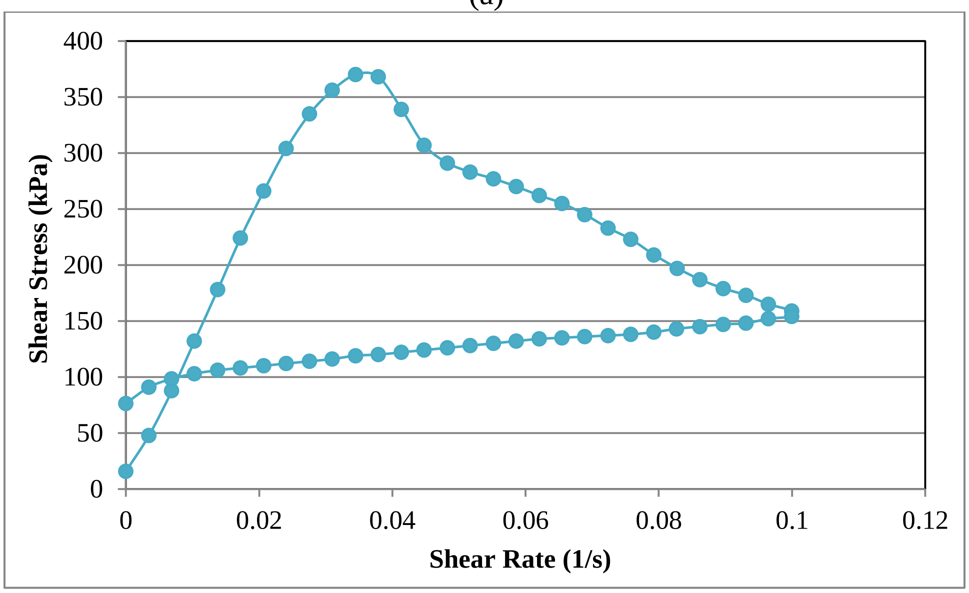


(b)

Figure 21(a) presents the hysteresis loops for the conventional asphalt binder at 5°C. As shown in this figure, the sample failed during the experiment as demonstrated by the distorted shape of the hysteresis loop. The distorted hysteresis loop criterion was suggested in the literature to assess the failure of HMA in fatigue tests (Al-Khateeb and Shenoy 2003). This trend was also observed when the maximum shear rate was reduced to 0.1 s^{-1} for the binder blend with 20% RAS from tear-off (T20), see figure 21(b). These results are possibly due to the brittleness of the binder and the negligible time-dependent viscoelastic behavior at low temperature. It may also be concluded from these results that the thixotropy effect will not be significant at low temperature as it is a typical manifestation of viscoelastic behavior of non-Newtonian fluids (Shu-Xin and Chuan-Jing 2006).



(a)



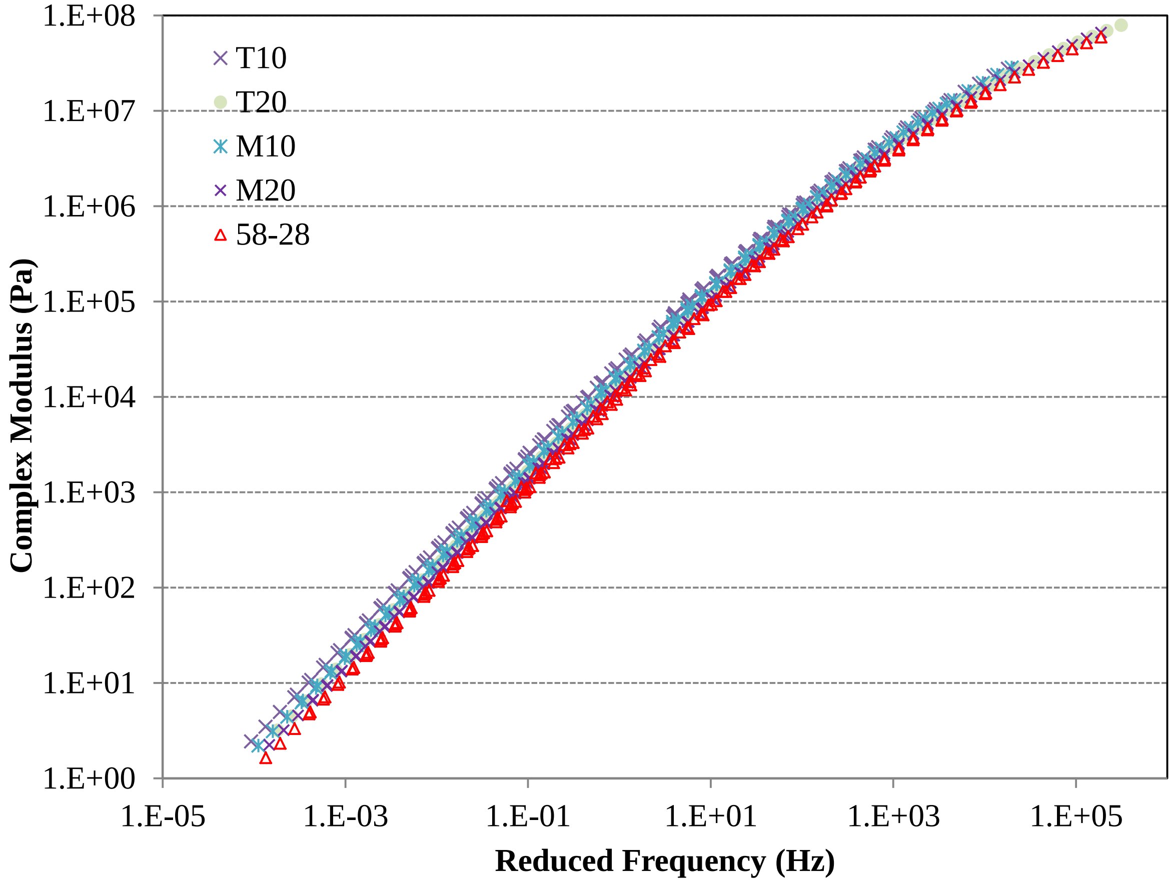
(b)

Figure 21. Hysteresis Loops at 5°C for (a) Control 52-28 (Cycle 2) and (b) RAS-Modified Asphalt Binders (Cycle 5)

4.6 Dynamic Mechanical Analysis

In order to analyze the effects of RAS on the behavior of the binder at intermediate and high service temperatures, the dynamic mechanical functions obtained from DMA were shifted in the form of master curves of G* and δ according to the time-temperature superposition principle (TTSP). The response curve at 25°C was considered the reference temperature, and all isothermal segments were shifted along the frequency axis to obtain a unique smooth curve. The

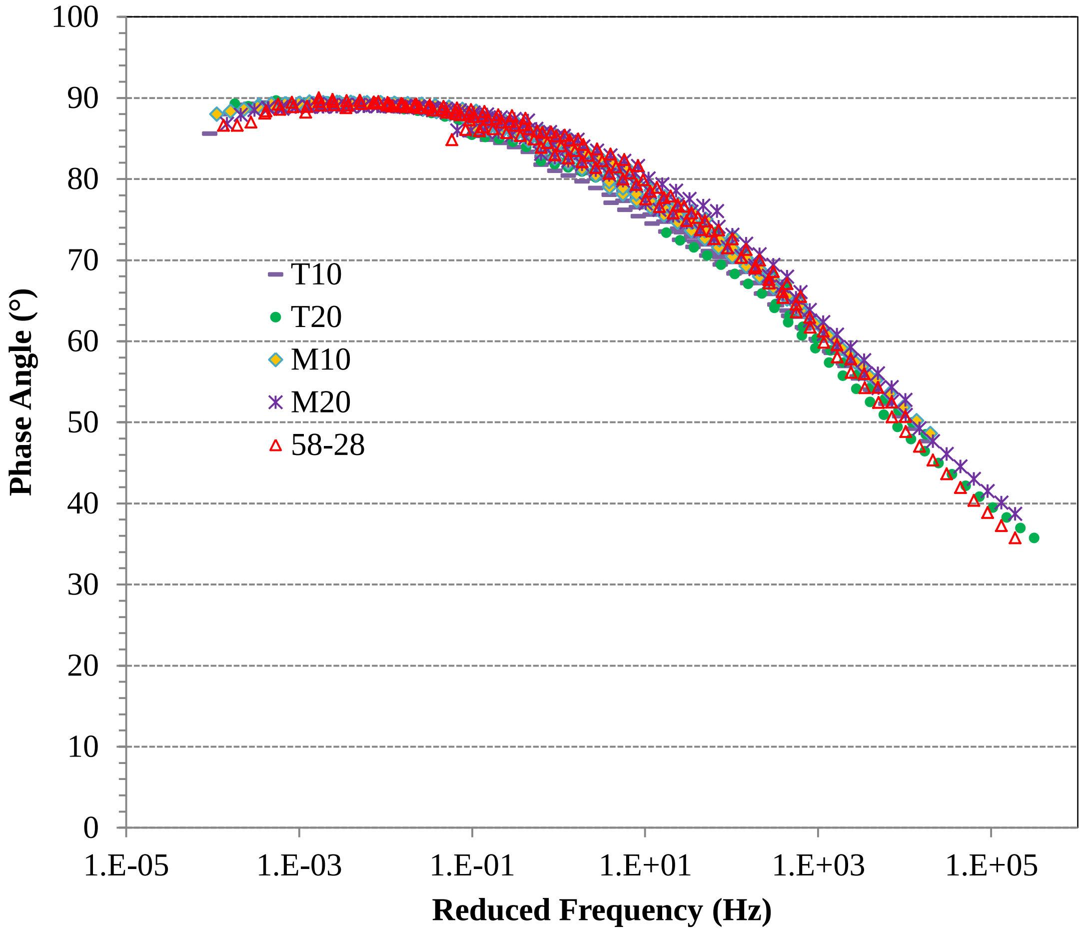
G* and phase angle master curves for the base and RAS-modified asphalt binders are shown in figure 22 (a and b). As shown in these figures, it is difficult to identify the effects of RAS in logarithmic plots of dynamic mechanical functions that extend over several decades of frequencies. Therefore, isochronal plots of the complex modulus and phase angle versus temperature were developed at 0.1, 11.3, and 100 Hz.



(a)

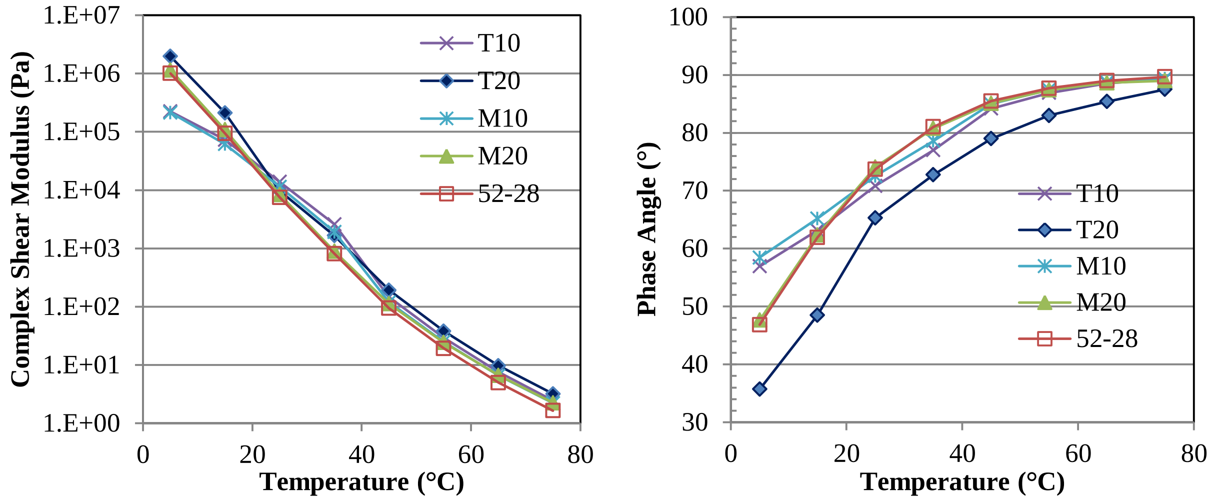
Figure 22. Dynamic Complex Shear Modulus and Phase Angle Master Curves for Straight and RAS-Modified Binders

(Figure cont'd.)

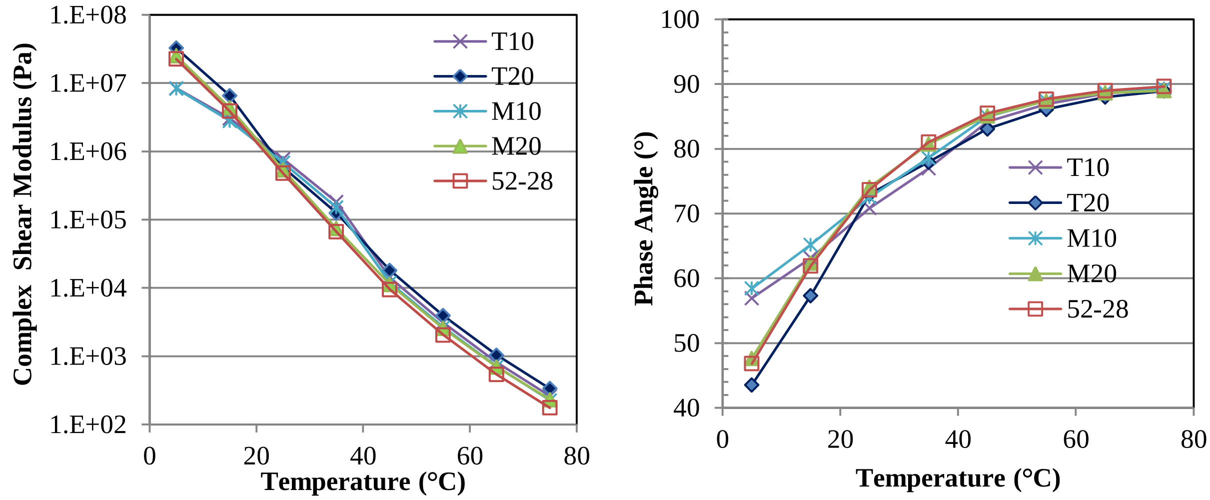


(b)

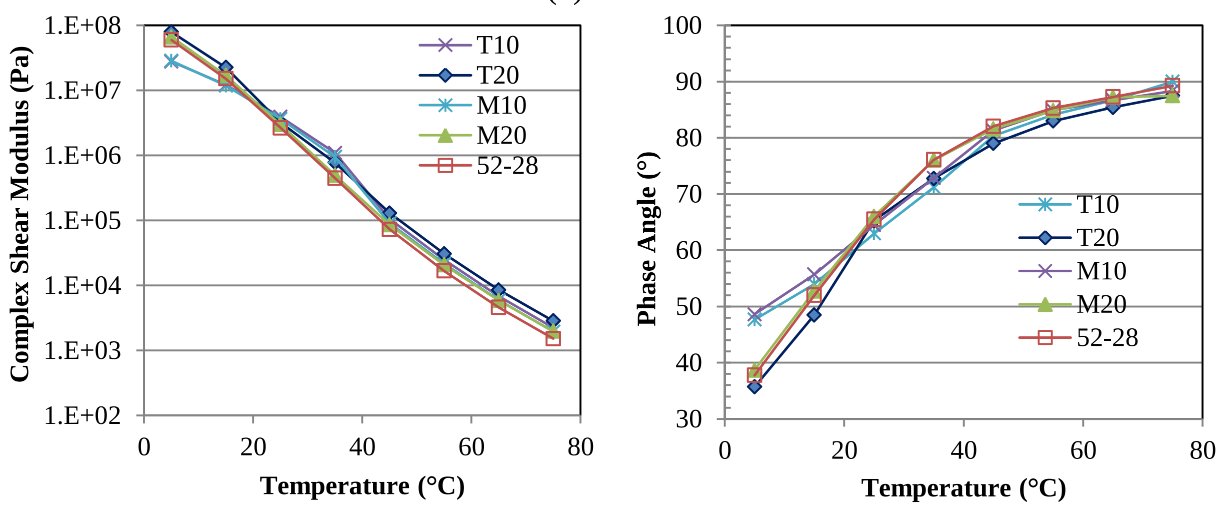
Figure 23 presents the isochronal plots of complex modulus and phase angles versus temperature at 0.1, 11.3, and 100 Hz. The addition of RAS caused a minor increase in the complex shear modulus at high temperatures, which may enhance the mix performance against permanent deformation. However, the phase angle did not follow a consistent trend with a decrease in phase angle at low temperature for the binder blends prepared with 20% tear-off RAS (i.e., T20) and an increase in phase angle at low temperature for the binder blends prepared with 10% RAS (i.e., T10 and M10). One may also note that there were no apparent differences between the binder blends prepared with tear-off shingles and those prepared with manufacturer waste shingles.



(a) 0.1 Hz



(b) 11.3 Hz



(c) 100 Hz

Figure 23. Isochronal Plots for Straight and RAS-Modified Binders

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary and Conclusions

The recycling of asphalt shingles in HMA is a very valuable approach for technical, economical, and environmental reasons. However, it is important that the influence of the recycling process is evaluated and quantified. Conventional practices of dry blending tear-off asphalt shingles with the aggregates before the asphalt binder is added to the batch are often criticized due to the large variability observed in the asphalt content of asphalt shingles and that the final PG grade of the binder is not known. The objective of this study is to introduce a new approach to recycle asphalt shingles in asphalt paving construction. In this method RAS is ground to ultra-fine particle sizes and blended with asphalt binder through a wet process then evaluate the rheological and thixotropic behaviors of shingle modified asphalt binders prepared using the wet process. In the proposed wet process, the ground recycled material is blended with the binder at high temperature prior to mixing with the aggregates. The proposed wet process allows for a better control of the chemical and physical reactions taking place in the binder blend.

In this study, two unmodified binders that are classified as PG 64-22 and PG 52-28 were blended with two contrasting sources of RAS at a modification level ranging from 10 to 40% by weight of the binder. The use of RAS modification through the proposed wet process was successful in the laboratory. Also the effects of shingle content and type, originating from tear-off and manufacturer waste sources, were investigated. The influence of adding ground shingle on the binder thixotropy at low, intermediate, and high temperatures was also evaluated in the laboratory using the hysteresis loop method.

Based on the results of the experimental program, the following conclusions may be drawn:

- Results of rheological and stability testing indicate that RAS can be used by proposed wet process at a modification content of 20% or less. The use of RAS modification through the proposed wet process would generally improve or not influence the high temperature grade of the binder. An optimum shingle content may be identified that will improve the high temperature grade without influencing the low temperature grade of the binder (e.g., 52T20 and 52M20 with a final PG grade of 58-28).

- Wax crystals ranging from 4 to 8 microns in size were successfully detected using Confocal Laser-Scanning Microscopy. A greater concentration of wax crystals was detected in the air-blown asphalt binder used in shingle manufacturing than in the soft PG 52-28 binder. However, wax crystals were not detected in the RAS-modified binder, which may indicate that the wax crystals were absorbed by the RAS binder.
- Results of HP-GPC showed that the binder in RAS had a high content of HMW. The proposed wet method of modification caused a slight increase of the HMW content in the prepared blends especially at high content of RAS modification.
- The use of RAS from tear-off and manufacturer waste shingles resulted in an increase in viscosity ranging from 3 to 130% when compared to the base binder. The increase in viscosity was proportional to the RAS content with greater increase at a RAS content of 30%. The increase in viscosity for the blends prepared with RAS from tear-off was greater than for the blends prepared with RAS from manufacturer waste. This may be due to the loss of light components in RAS from tear-off during service. Still this increase is not affecting the workability of asphalt.
- The temperature susceptibility of the binder in the range from 95 to 135°C decreased with the use of RAS. This may be due to the granules present in the RAS that are typically temperature-inert. However, in both cases, the change in the binder VTS with the use of RAS was minor.
- Thixotropy and shear thinning were observed concurrently in the asphalt binder blends at 25°C. RAS-modified asphalt binders showed greater susceptibility to thixotropy than the base binder. Thixotropy increased with the increase in RAS content for both tear-off and manufacturer waste shingles. For all asphalt binder blends, thixotropy decreased with cyclic loading.
- For all asphalt binder blends, thixotropy effects were negligible at high and at low temperatures.

This study represents a first step towards evaluating the proposed wet process to recycle waste asphalt shingles in asphalt paving construction. Based on the results of this study, further research is recommended to evaluate the design and performance of asphalt mixture prepared with the proposed approach. Research is also needed to consider other asphalt binder sources including polymer-modified binders (e.g., PG 52-46 or PG 58-34).

5.2 Future Research and Recommendations

- In a future study, application of RAS using the wet process in asphalt mixture should be evaluated; mixture tests can support the rheology findings presented in this study.
- In order to evaluate the chemical effects of RAS on binder, blends should be analyzed by the SARA system (ASTM D 4124; Separation of Asphalt into Four Fractions; asphaltenes, saturates, naphthene aromatics, and polar aromatics). SARA can accurately define the amount of asphaltene and maltenes in the binder blends.
- In this study, limited sources of shingle were mixed with the binder. More sources need to be investigated in order to determine the effects of environment on asphalt content in RAS and its quality.

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APPENDIX A: EXTRACTION SYSTEM

Figure A1. Reflux System (Left) Rotaevaporator (Right)

APPENDIX B: SAMPLE PREPARATION AND TESTING EQUIPMENTS

Figure B1. Ground Tear-off Sample

Figure B2. High Shear Mixer Mixing RAS and Control Binder On Top of a Hot Plate

Figure B3. Cigar Tube Preparation

Figure B4. Anton Paar MCR 301 and 302 Used for Rheology Tests

APPENDIX C: PARTICLE SIZE ANALYSIS

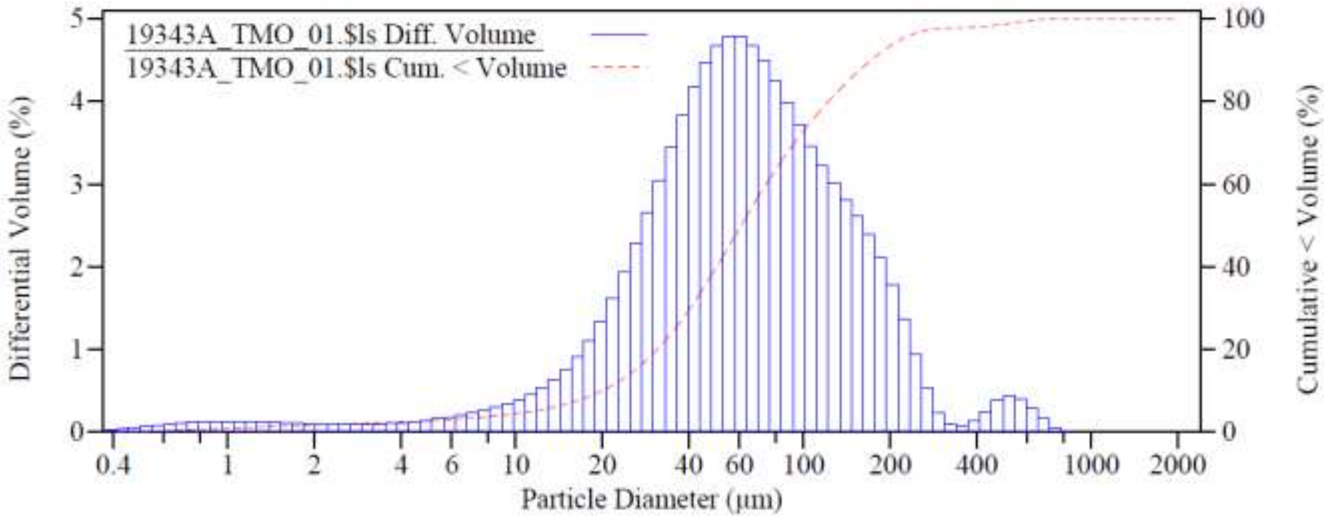

Figure C1. Particle Size Analysis of Tear-off Sample

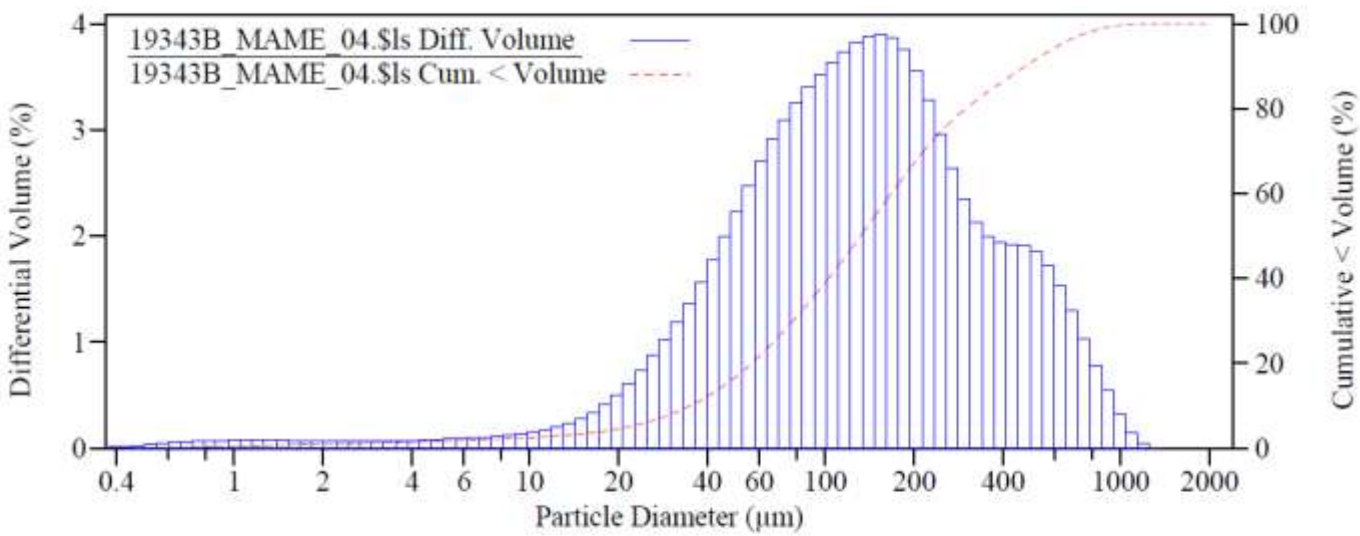

Figure C2. Particle Size Analysis of Manufactured Sample

VITA

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